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AND NATURAL GASOLINE MANUFACTURER

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November
1933

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ANNUAL MEETING

DIVISION OF REFINING—AMERICAN PETROLEUM INSTITUTE

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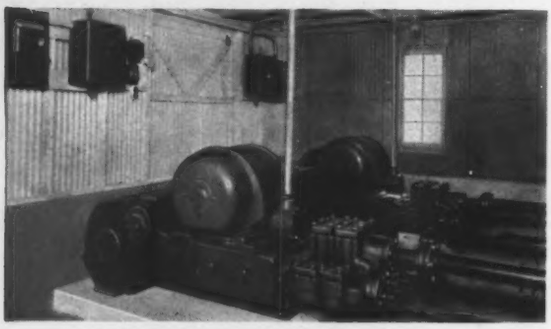
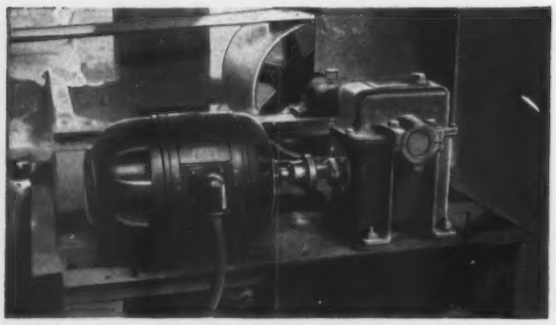
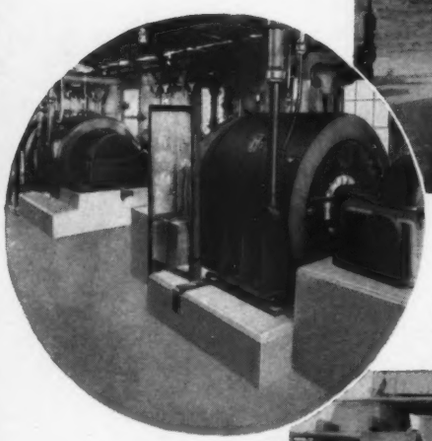


Two G-E splash-proof motors driving gathering pumps

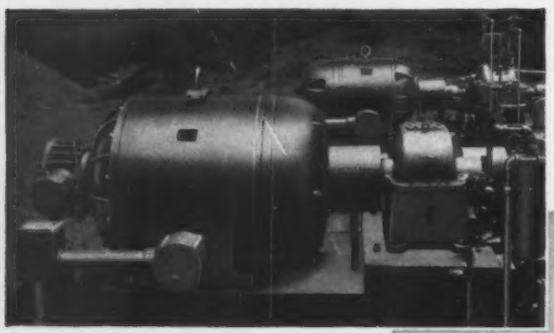


G-E totally enclosed, fan-cooled, squirrel-cage motor driving conveyor under fuller's earth burner

Outdoor installation, G-E explosion-proof motor driving oil-well pumping unit

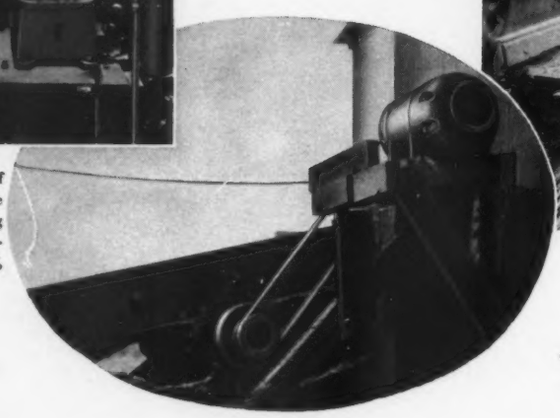


Two G-E totally enclosed, fan-cooled, inert-gas-filled synchronous motors in casing-head-gas gathering plant



Foreground, G-E 100-hp. synchronous motor of Class I, Group D, construction driving gasoline and reflux pumps through G-E speed-increasing gear; background, standby unit with G-E 75-hp. totally enclosed, fan-cooled, explosion-proof, squirrel-cage motor. Outdoor installation.

G-E single-phase explosion-proof motor driving mud screen, outdoors



G-E totally enclosed, fan-cooled, squirrel-cage motor driving mud screen, outdoors

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GENERAL ELECTRIC

REFINER
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NOVEMBER, 1933
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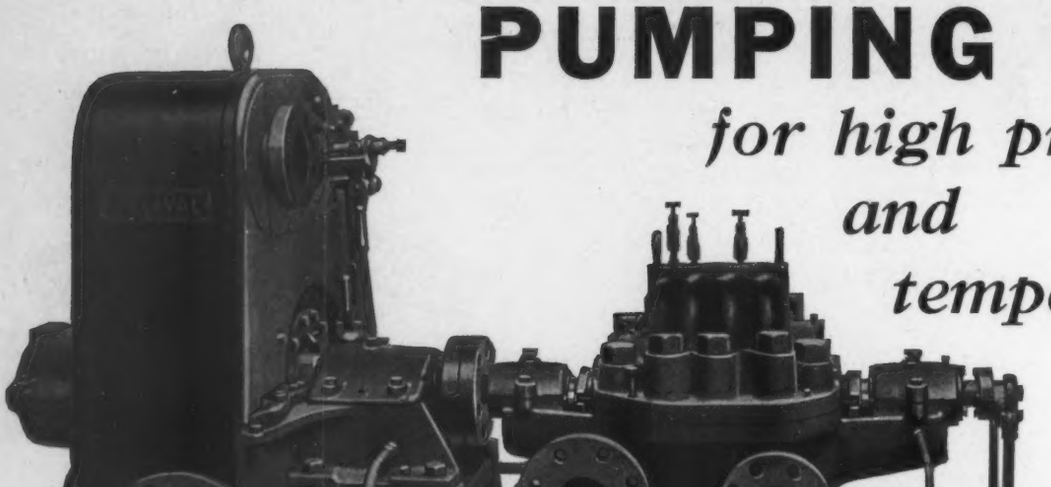

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VOLUME 12, No. 11 NOVEMBER, 1933

Solvent Refining . . .

BECAUSE of the active interest in solvent refining, the American Petroleum Institute Division of Refining at the semi-annual meeting last June in Tulsa, devoted a session to the study of such developments. Again, at the recent annual convention in Chicago the division devoted two sessions to presentation and discussion of eight papers dealing with solvents and solvent processes. Several of these important papers are presented in this issue.

These papers give details of developments more recent than data available at the mid-year meeting and some of them go into details of construction and operating costs, as well as presenting results of technical studies and characteristics of both the various solvents used and the finished products secured by such processing.

Among the processes described are the Edeleanu, using sulphur dioxide, or a mixture of sulphur dioxide and benzol; the Nitrobenzine process employed by The Atlantic Refining Company; the Chlorex process, using dichlorethyl ether, developed by Standard Oil Company (Indiana); the Duo-Sol process (employing two immiscible solvents) developed by Max B. Miller Company; the Phenol process, developed and used by Imperial Oil Company of Canada; the Furfural process developed by The Texas Company; the process developed by Foster Wheeler Corporation reported to use crotonaldehyde as a solvent, and the use of propane in dewaxing as reported by Union Oil Company technologists.

Some of these processes were also discussed at the mid-year meeting at Tulsa, where in addition reports were presented dealing with processes such as the Govers process, using acetone and benzol for dewaxing, as developed by Indiana Refining Company (The Texas Company subsidiary); also Standard Oil Company's (Indiana) process using propane solution with self refrigeration was discussed, as was the S-N Tri process (trichlorethylene) developed by the Separator-Nobel Company of Sweden, and now under investigation semi-commercially by Standard Oil Company of New Jersey. These papers appeared in the June, 1933, issue of The Refiner.

In addition to the solvents enumerated above which are now applied commercially in the industry it is said that there are about 50 more solvents which will be brought to the attention of the industry within a short time. Several of the larger refining companies have developed their own solvent processes, are using them in their own refineries and planning additional installations. Other large companies are installing processes licensed by various consulting or process manufacturing concerns. At present their application is under way at refineries not possessing supplies of paraffin base crude such as Pennsylvania grade. The development of these processes makes possible close approach to specifications of lubricants produced from Pennsylvania grade crude. In addition, work now under way indicates that the application of such processes to Pennsylvania crude lubricating oil fractions will improve those oils much above present standard.

It is the opinion of most refining technologists that the various solvent processes now available, and new developments to be announced soon, all employing virtually the same principle of selective solvent action on lubricating hydrocarbons but varying somewhat in their details of application, will no doubt revolutionize the science of lubricant manufacture. The present active interest in such processes, the present investigations being carried out, and the present actual installation work in this field certainly points in that direction.

The presently looming "quality race" in the refining industry will be in connection with the improvement in viscosity-temperature curve of lubricating oils, and result in a general raising of viscosity-index of commercial lubricants, lessened oxidation with greater resistance to sludging, lower carbon residue, and improved pour points, flash and fire.

Solvent Extraction *and It's* Application

JOHN W. POOLE and J. M. WADSWORTH
Foster Wheeler Corporation, New York, N. Y.

THE paper is designed primarily for the refinery executive and the refinery chemist whose activities have not been directly concerned with solvent-extraction methods.

Solvent extraction, as generally regarded, falls into two classifications—the first of which, true solvent extraction, is useful for the production, from comparatively low-grade raw material, of oils of higher A.P.I. gravity and viscosity index and of lower carbon residue and sulfur. From neutral oils it is possible to manufacture, without acid or clay treatment, oils as light in color as those called for in the most stringent specifications. Residual oils require either acid or clay for proper color reduction. The second type of solvent extraction or solvent dilution consists of adding to a wax-bearing oil a non-viscous solvent miscible with the liquid portions and capable of reducing their viscosity in order that solid constituents, which remain essentially unaffected by the solvent, may be more easily removable. Comparisons of oils treated by solvent and by conventional methods are given. A flow sheet of the Foster Wheeler process is included.

This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933.

SOLVENT-EXTRACTION processes may be classed in two groups: the first we might call solvent dilution; the second we could call true solvent extraction. In the first class come all processes which have been prominent in recent years as new methods for dewaxing neutral and residual lubricating stocks. To the minds of many people these solvent-dewaxing processes actually are true solvent-extraction methods. In order to clarify this and distinguish between the two classifications of solvent refining we may consider the theory underlying organic solutions.

SOLVENT-DILUTION EXTRACTION

In general, organic solutions are different from inorganic solutions in that two different types of solution usually exist. For instance, if two liquids are mixed together in a suitable container, one of two things may occur. First, as we add the second liquid to the first, we may find that the second liquid becomes dissolved completely, forming only one phase—regardless of the extent to which this second material is added. On the other hand, we may find, when the second liquid is added to the first, that for an interval the same phenomena will occur, but that presently the first material becomes saturated with respect to the second, and that further addition of solvent results in the formation of a distinct and separate second phase. In many cases this phase is discernible in that the phases are of different specific gravity, the heavier tending to collect in the lower portion of the container. In such cases a sharp interface is usually discernible, especially if the two phases are not identical in color. In the case of organic solvents and those mixtures of hydrocarbons which are derived from petroleum to form the bases for lubricating oils, both of the above-mentioned cases may occur.

With solvent-dilution dewaxing processes, the condition is essentially that covered by the first of the above cases, i.e., only one true solution is formed. To this, the obvious reaction might be that two phases are formed; otherwise it would seem impossible to remove the wax from the oil in order to effect the object of the process. What actually occurs is, however, single phase, since one of the phases occurring in a solvent process for dewaxing may not properly be considered a solution. It is believed that the actual occurrence is as follows:

When such solvents as acetone, butyl alcohol, ethylene dichloride, or any one of the solvents capable of effecting the desired results is added to a wax-bearing lubricating stock, this solvent goes into solution in the liquid portion of the stock. It is an established fact that hydrocarbons upon passing from the liquid state to the solid state become far less miscible with organic solvents, regardless of the solvent. In many cases this reduction in miscibility may be insufficient to avoid liquefaction of the solid hydrocarbons in the mixture of hydrocarbons and solvent. Such solvent would, in consequence, be useless for dewaxing processes. On the other hand, there is unquestionably a vast number of organic liquids which are highly miscible in liquid hydrocarbons but which have extremely low miscibility with any hydrocarbons present that are below their melting points. This class of solvents may be said to be uniformly useful as dewaxing solvents—at least from

the theoretical viewpoint. Their only purpose when added to the wax-bearing stock is that of diluents. Since they have little affinity for the paraffin waxes which are solid at the temperatures under which the process is operated, this wax will remain practically unaffected by their presence.

Moreover, if we choose one of the solvents highly miscible with the wax-bearing stock and which possesses considerable fluidity, the presence of this solvent will so reduce the viscosity of the liquid portion of the process material that the solid particles remaining essentially unchanged may be more readily removed from the solution. In short, the problem is purely one of mechanics.

Therefore, to sum up briefly, this type of solvent refining merely consists of diluting a mixture already comprising solid paraffins and liquid oil. The addition of the solvent merely serves the purpose of increasing the fluidity of the liquid portions so that these portions may be more easily strained, filtered, or centrifuged from the solidified wax. It is our belief that processes for precipitation of tar come within this classification of solvent separation, e.g., the laboratory method for the determination of tar, consisting of diluting the liquid constituents of the sample with petroleum ether so that they may be filtered from the semi-solid tar.

To illustrate solvent-dilution extraction methods we refer to Figures 1 and 2. Figure 1 represents a certain

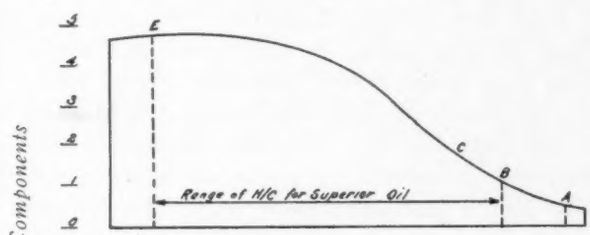

FIGURE 1
Compositional Characteristics of Pennsylvania Lubricating Stocks.

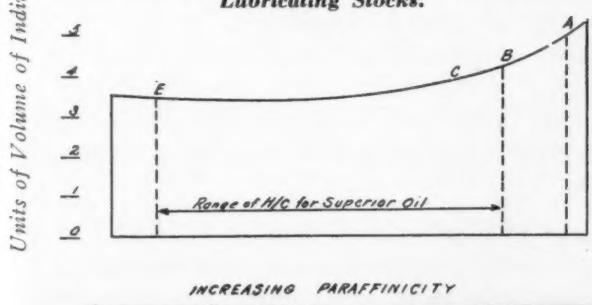

FIGURE 2
Compositional Characteristics of Mexican Lubricating Stocks.

sample, for instance, one barrel of Pennsylvania stock. Figure 2 represents the same amount of an oil of the same boiling range from a Mexican source.

For simplification assume that the components of these two oils vary in hydrogen-carbon ratio, but not in boiling point. Those portions of each oil which are represented by the area at the extreme left of Figures 1 and 2 possess the highest possible hydrogen-carbon ratio. Those portions at the extreme right possess the lowest hydrogen-carbon ratio of all. The latter also contain an appreciable proportion of the sulfur and oxygen compounds, together with such materials as will form sludgy or gummy matter. All points intermediate between these two extremes will also be intermediate in hydrogen-carbon ratio.

Now, since the value of hydrocarbons as lubricants varies in more or less the same way as does hydrogen-carbon ratio, it will be true that as we move from right to left, we pass hydrocarbons of constantly increasing value, until we reach the point **E** in either figure. At this point the percentage of hydrogen has become so high, and the internal structure is such, that the hydrocarbons become solid paraffins; and, because they are solid, they must be rejected.

In both Figures 1 and 2 the area to the right of point **A** roughly represents such portions as present-day processes of acid and fullers-earth treatments tend to remove. The areas between **A** and **B** represent constituents of low hydrogen-carbon ratio, relatively high in sulfur, and which are scarce in Pennsylvania oils. It is due to their presence that Mexican lubricating and similar oils are often considered inferior to Pennsylvania oil. These materials are not readily removable by sulfuric acid or clay. Whereas they do exist in Pennsylvania oil, their volume is relatively unimportant, and is more or less lost in the large percentage of "paraffinic" material. We may say that the areas between **E** and **A** in the two figures represent the quantities and indicate the natures of the lubricants which could be produced from the respective sources by usual methods.

For this type of separation it is not necessary to choose a truly selective solvent. The only requirement is that we choose a solvent which is highly miscible with such materials as are represented in these two figures by the areas to the right of point **E**, and with such portions of the material to the left of point **E** as are liquid at the temperature of operation, but which very sparingly dissolves the solid materials to the left of point **E**. In consequence, as the temperature is reduced, more and more of the material to the left of point **E** will be reduced to temperatures below its melting point, and will become through this change of state insoluble in the diluting solvent which has been chosen. At the proper temperature this solid material may be centrifuged or filtered from the mixture of liquid hydrocarbons and dissolved solvent, thereby effecting a separation between solid and liquid components. That this is the type of action which actually occurs is borne out by a considerable quantity of unpublished data which is in our possession.

For instance, if such a mixture is carefully filtered, the filtrate will be obtained in a clear and transparent condition—showing that no solid particles

exist. Furthermore, after the filtrate has been freed of solvent, it has been found that, regardless of the solvent employed (provided wax of the type separated was sparingly soluble in this solvent), the resulting oil will possess a pour point essentially the same as the temperature of filtration. This point has been established with a considerable variety of solvents of all types including butyl and amyl alcohols, paraldehyde, ethylene dichloride, trichlorethylene, nitrobenzene, methyl ethyl ketone, bromobenzene, and many others.

TRUE SOLVENT EXTRACTION

The second type of solvent extraction, viz., true solvent extraction, may be said to be the direct opposite of the case just described in that the so-called solvent actually serves as a solvent rather than as a solute. In such a case it is necessary to choose a solvent which will not dissolve to a high degree in the "paraffinic" material of the charging stock. For example, if this material will become saturated upon the addition of 20 percent of solvent, there will, at this point, develop the second or true solvent phase. In this latter phase there will, provided the solution is a suitable one, become dissolved certain portions of the original stock. As greater and greater amounts of solvent are added, the percentage of the original material which is dissolved will increase. When the total of the solvent selected has been added and thoroughly mixed with the charging stock, and the mixture allowed to settle, these two phases may be separated one from the other by mechanical means such as decantation. When this separation has been completed, there will be contained in one vessel the undissolved portion of the original material plus 20 percent of solvent. In the other vessel there will be contained the remaining portion of the solvent, together with such portions of the original stock as have proved to be soluble in it. If the solvent is removed from both of these separated phases, obviously there will be segregated portions of the original raw material in separate containers. If, then, the solvent has been properly chosen, i.e., it possesses the quality of "selectivity" to a high degree, these two fractions will differ considerably from each other. Furthermore, the difference will be as follows:

The undissolved portion coming from the so-called oil phase will be constituted primarily of the more "paraffinic" materials which were originally present. The fraction produced by removal of the solvent from the true solvent phase will consist of materials highly "naphthenic" in nature or, in other words, of material of low hydrogen-carbon ratio, unsaturated compounds, hydrocarbon derivatives containing oxygen, nitrogen, and/or sulfur, etc.

In general, when a raw hydrocarbon material is mixed with a solvent, and there results from this mixing a segregation into two phases, each phase of which contains both oil and solvent, it will be found that the above types of raffinate and extract will be produced. However, the degree to which the raffinate will be "paraffinic" and the extract "naphthenic" will depend upon that quality which we choose to call "selectivity." If the solvent chosen is highly selective, the difference between these two materials will be great. If the selectivity is poor, the difference will be small; and, obviously, we may say that intermediate degrees of selectivity may be measured according to the degree of difference between the two products which are produced.

The solvents used by Foster Wheeler Corporation in its work possess selectivity to a very high degree which, when interpreted in terms of Figures 1 and 2, means that they possess an unusual ability progressively to dissolve hydrocarbons. They will most easily dissolve material at the extreme right of Figures 1 and 2; and, if we so desire, we can control them so as to dissolve material only up to point **A**, thereby taking out essentially the same material as is now removed by acid and clay. On the other hand, it is possible to control solution in such a manner as to dissolve all material from the extreme right to practically any point to the left, for instance up to point **B**. The solvent containing the dissolved material can be separated, as we have already shown from the portion remaining undissolved—leaving behind a lubricant which petroleum chemists cannot tell from that manufactured from Pennsylvania oil. In fact, if we so choose, we can produce in this manner an oil even more truly "paraffinic" than Pennsylvania oil merely by removing the material between **B** and, for instance, **C**. The point where we stop is purely one of economics, and each refiner will naturally decide that point for himself. The important fact is that it is possible to take raw material of which an abundance exists, and secure from it a high percentage of oil as good as, or even better, than the highest quality Pennsylvania product. It is also feasible to remove from a high-sulfur oil a larger percentage of the sulfur and carbon-producing compounds. This method of solvent fractionation is of the type representing true selective solvent separation.

CHOICE OF SUITABLE SOLVENT

A solvent must meet certain requirements if a resulting process is to be effective and economical. Nine such requirements are listed below, each of which is an essential for satisfactory operation. Simultaneously to satisfy all nine of these points is not a simple matter, especially since certain ones alone are satisfied by few organic solvents. These characteristics are so rare, in fact, that when viewed in connection with the other essentials, it is surprising that such things as satisfactory solvents exist at all. The nine prerequisites are as follows:

1. The solvent must possess much greater affinity for "naphthenic" material than the affinity which

the paraffin compounds, comprising the oil phase, will possess in order that comparatively small volumes of solvent will be required. This means that the distribution ratio of "naphthenic" matter between solvent and "paraffinic" compounds will be high. Not only is a high distribution ratio important in order to reduce the volumes of solvent required, but also to reduce the number of contact stages in a commercial plant.

2. The solvent must possess low solvent power for "paraffinic" material, in order that the percentage of recovery of such material may be high.

3. The solvent must be relatively insoluble in the "paraffinic" material. Obviously, such solvent as becomes dissolved in the oil phase will not be available for extraction from this phase. If extreme volumes are necessary to effect saturation of the "paraffinic" phase, a process using such a solvent will be forced to handle excessive volumes of material—thereby requiring large and expensive apparatus. Greater capital outlay will be necessary, and fuel costs for distillation recovery of solvent will also be increased.

4. The solvent under operating conditions must be chemically stable. If not stable, there will be an expensive loss of solvent due to its self-destruction. Furthermore, if chemically reactive, undesirable changes may be brought about in the final products.

5. The solvent should be relatively low boiling, in order that it may be easily and more completely recoverable. In dilute solutions the partial vapor pressure of a substance decreases directly with decreasing concentration. Certain solvents which possess high boiling points become very difficult to recover beyond 95 percent, except under high temperatures. Necessity for high temperatures may result in degradation of the oil.

6. The solvent process must be operable under normal conditions of temperature and pressure. If the process is operable at normal atmospheric temperatures, the expenses of refrigeration or heating will be avoided. In order to operate at normal atmospheric pressure, the solvent must boil above the temperature of operation. From Paragraph 5 it follows, therefore, that the boiling range of suitable solvents is limited, probably between 90° F. and 225° F. We believe that the ideal solvent should boil in the neighborhood of 140° F.

7. The solvent must be non-toxic; i.e., it must exert no undesirable pathological effects on the operators, or be destructive of animal tissue.

8. The solvent should be relatively low in price. Regardless of whether the solvent is highly recoverable in any commercial plant, there must, of necessity, be losses due to leakage—and even due to the difficulty of recovering the last trace of solvent from the process products. In theory, the latter loss can be made very low—although it may be demonstrated, through the laws of vapor pressure, that it may never be completely eliminated.

9. In order to avoid high investment costs and maintenance charges, the solvent should not be corrosive to ordinary construction materials.

FOSTER WHEELER PROCESS

The first stages of development of the Foster Wheeler process were carried on at the Massachusetts Institute of Technology, and later continued in the Foster Wheeler Research Laboratory. The solvents used in this process comprise a certain definite class of chemical compounds, varying slightly in general chemical characteristics but appreciably in volatility. The two solvents that we have most highly developed are crotonaldehyde and acrolein which, for convenience, are designated as solvent "A" and solvent "B," respectively. Of course, the commercial grade of solvent will differ somewhat in characteristics from the chemically-pure compounds; these differences have not proved detrimental. We have, in fact, used commercial solvent in all of our recent development.

The physical characteristics of these two solvents, as taken from the *Handbook of Chemistry and Physics*, are as follows:

Crotonaldehyde (solvent "A"): C_3H_5CHO

Molecular weight	70.07
Specific gravity	0.859 at 14° F.
Melting point	....*
Boiling point	104—105†

Acrolein (solvent "B"): $CH_2CH.CHO$

Molecular weight	56.05
Specific gravity	0.84
Melting point	....*
Boiling point	52.4° C.

*From our observation we know the melting point to be less than 0° F.
†We believe this somewhat high, and that the boiling point is around 103° C.

Since solvent "B" boils considerably lower than "A," it is the more attractive from the volatility standpoint; solvent "B" has not, however, been as much used in our commercial development as "A." The latter possesses distinct commercial uses other than solvent extraction; and is, in consequence, much more easily and cheaply obtainable. Although boiling at a higher temperature than solvent "B," it does, nevertheless, approach more closely in volatility characteristics the specifications of an ideal solvent than do other groups of selective solvents which we have investigated.

The following will show how well solvent "A" satisfies the nine requirements previously mentioned as requisites for a suitable solvent. In general, solvent "B" and other solvents in this group have similar physical characteristics.

1. Solvent "A" possesses greater affinity for "naphthenic" material than any other selective solvent which we have investigated. At a temperature of 68° F., the distribution ratio of "naphthenic" material with respect to solvent and a pure Pennsyl-

vania neutral oil varies between four and 20, depending on the nature of the "naphthenic" materials used as standards.

2. At 68° F., solvent "A" only sparingly dissolves Pennsylvania neutral or bright stocks.

3. Solvent "A" dissolves in Pennsylvania bright stock to the extent of 10 percent to 15 percent; in Pennsylvania neutral, to approximately 16 percent.

4. Solvent "A," provided oxygen is excluded from the system, is very stable. This will be indicated by the degree of recovery which we have obtained as hereafter discussed under Paragraph 5.

5. In quantitative runs conducted under atmospheric pressure for the purpose of determining recoverability of solvent "A," we have been able to recover as high as 99.9 percent of the solvent at maximum distilling temperatures less than 350° F., and 10 percent of steam. A similar recovery has been secured by using an absolute pressure of 21 mm. and the same maximum temperature of distillation using no steam. Figure 3 is the vapor-pressure curve of a two percent solution of solvent "A" in a 250-viscosity-at-100° F. neutral oil from which it is possible to calculate the distillation conditions necessary to reduce the solvent loss in the oil to the desired minimum.

6. Up to the present time we have never encountered a lubricating stock which required contact temperatures less than 60° F., and for many stocks efficient extractions can be performed at temperatures as high as 90° F.

7. Solvent "A," although possessing a disagreeable odor, is neither toxic nor destructive to animal tissues. The senior author and his assistants have worked more or less continuously in an atmosphere containing this solvent, and have never suffered any ill effects.

8. Solvent "A" is moderate in price. At present it is available at approximately 75 cents per gallon, and it is expected that the price will further decrease with increased demand.

9. Solvent "A" under conditions of operation is not corrosive. The pure solvent may be shipped or stored in steel containers. In the pure state the solvent does possess a peculiar ability to be slightly corrosive to brass or copper. However, this corrosive power is lost when as little as two percent of hydrocarbon material becomes dissolved.

Table 1 gives the results which have been obtained on various types of crude lubricating distillates and residual stocks. To a considerable degree this tabulation is self-explanatory. Run 1 shows the original stock and the products resulting from a 120 percent, three-stage counter-current treatment of a Mid-Continent reduced pressed distillate. This material contained appreciable residual matter—probably as entrainment—and was, in consequence, very dark in color. The primary product was produced by treating the raffinate with 2½ pounds of acidified "filtrol." The secondary product was distilled from the extract, and then treated with 10 pounds of 1.84-acid for color. For comparison, the original stock was treated and filtered in the conventional manner with 10 pounds of 1.84-acid and five pounds of "filtrol" to produce an oil with a color equal to the finished raffinate. This comparison shows the marked improvement in gravity, viscosity index, and carbon residue secured with solvent "A."

However, the charging stock for Run 1 was not prepared especially for solvent extraction. Since there are incorrect ways and only one correct way of operating the process, there are certain precautions involving very little extra care and no additional

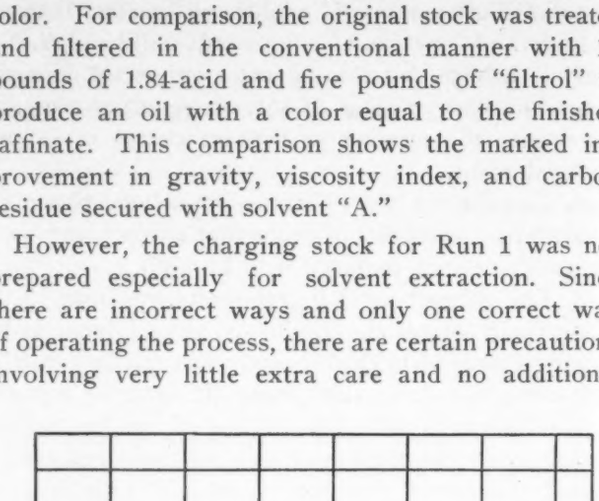

FIGURE 3
Vapor-Pressure Curve for 200-Viscosity Neutral Oil Containing 2 Percent by Weight of 2 Percent Bottoms of Commercial-Grade Solvent "A."

expense which must be regarded as if a distillate is to be efficiently handled by a solvent process.

Runs 2 and 3, on lubricating distillates produced from the same crude as was the raw material used in Run 1, show how color and carbon residue can be improved by properly processing the original stock. In neither of these runs was it necessary to give any auxiliary treatment of acid or clay after the solvent process. Colors of the raffinate as obtained directly from these distillates were, respectively, 1¾-N.P.A. and 2¾-N.P.A., and the carbon residues were extremely low. It will be noted under Run 2 that the flash points of both the primary product and the

extract have been raised. This may be due to either one or both of two reasons. Since low-boiling constituents can be dissolved more easily than those boiling higher, raffinates are frequently produced possessing higher flash points than did the original oil. The extract generally possesses a flash point at least as low as original stock. In this particular run the extract appears to have been raised five degrees. This was probably due to a slight vaporization of hydrocarbon material at the time the solvent was removed, by distillation. With regard to the ability to secure raffinates direct from the process—meeting commercial color and stability specifications—we have consistently been able to produce such colors, and have been able to produce a raffinate of greater than 200 viscosity at 100° F., with colors as low as 1-N.P.A. With very light acid and clay treatment in addition, we have produced from such raffinates oils of color corresponding practically to blank on the N.P.A. colorimeter. Such oils generally possess a slight bloom.

We have not been able to treat residual oils to color without the subsequent use of some acid or clay. Nevertheless, we have been able greatly to reduce acid and clay quantities over conventional practice, and have reason to believe we can eventually reduce this auxiliary treatment to as little as a half pound of acid and five pounds of "filtrol," or equivalent, per barrel of typical Mid-Continent raffinate of 100-150 viscosity at 210° F.

Briefly, our accomplishments with cylinder stock are these: In every case we have been able materially to improve viscosity index, reduce carbon residue, and decrease specific gravity of products by solvent extraction and subsequent filtering, or combined acid and clay treatment, for final color. With yields of around nine-tenths of that by conventional methods we have increased viscosity indices as much as 30 points, decreased carbon residue by more than one half, and raised the A.P.I. gravity by three degrees. Runs 4 and 5 illustrate definite examples of treatment of cylinder stocks, where solvent extraction constitutes a step in the process.

In Run 4 two products were prepared from a Mid-Continent residuum. One was treated by conventional practice with 30 pounds of 1.84-acid per barrel for a sludge loss of approximately 28 percent. The other was subjected at 68° F., to 200 per cent two-stage counter-current flow of solvent "A." Extracts equal to 23 per cent of the charge were produced, being asphalts of extremely high dcutility. Also 8 percent of ceresin wax was centrifuged from the oil phase before the solvent was removed. The removal of ceresin was made possible by the increase of fluidity due to the presence of dissolved solvent (an example of solvent dilution). This partially-dewaxed raffinate was then treated with 10 pounds of 1.84-acid per barrel, equivalent to about 7 pounds of acid based on the original charge; resulting in the production of a stock for filtering equal to approximately 59 percent of the original charge, as against 72 percent in the previously-mentioned case of acid treatment alone. If we consider that this 72 percent yield includes 8 percent of ceresin (equivalent to that removed in the solvent step) we should have 64 percent filterable oil by the conventional method as compared to 59 percent by the solvent method. These two filterable oils were each contacted at a comparatively low temperature (400° F.), using 29 pounds of "filtrol" on the basis of the original charging stock. The results on these two oils are shown in Table 1. It will be noted, as in Run 2, that the flash of the raffinate was improved over the original stock. No mention has been made of viscosity indices on these oils, since the presence of wax crystals causes low-temperature viscosities to be inaccurate and, in consequence, similarly affects the viscosity index.

In the case of Run 5 the primary product was produced by the subsequent use of 10 percent of acidified "filtrol." The original stock could not be treated by acid to form a separable sludge. Quantities of clay of greater than 40 percent would have been necessary to produce the same color on the original stock as was produced on the raffinate with only 10 percent of "filtrol."

Runs 6 and 7 give partial data on the solvent treatment of certain California lubricating stocks which were not produced by the proper methods necessary to secure improved colors and carbon residue from solvent treatment alone.

Run 8 shows a California distillate produced when such precautions were regarded. We regret that we do not possess more complete data on California products, but it is evident from the tabulation that it is possible to produce oils of high viscosity indices from this source.

Due principally to a high yield of oil of considerably increased viscosity index, results from Mid-Continent stocks are generally more impressive than those from other sources. On the other hand, the Foster Wheeler Laboratories have found it possible to effect desirable results with practically all classes of oil. For instance, with oils from the Pennsylvania field, it is often possible to increase viscosity indices as much as 20 points without extracting more than 10 percent. Furthermore, the naturally high carbon residues of this type of oil will be considerably reduced by such treatment, and filtering costs will also be reduced. From oils highly "naphthenic," such as California stocks, it is possible to produce raffinates of greatly improved viscosity index—approaching, if necessary, that of Pennsylvania products. On raffinates from California neutrals the carbon residue is extremely low, providing the original distillation has been reasonably well done—usually being reportable as only "a trace." Yields are, of course, low.

Since solvent extraction is purely a separatory process, it is only possible to recover such highly "paraffinic" oil as originally existed. Furthermore, with treatment wherein it is necessary to use high percent-

TABLE 1

Comparison of Proportions of Original and Finished Oils Prepared by Foster Wheeler Solvent-Extraction Process Versus Conventional Acid-and-Clay Treatment

Material	Yield (Per cent)	Solvent (Per cent)	Temperature (Deg. F.)	Gravity (Deg. A.P.I.)	Flash Point (Deg. F.)	Fire Point (Deg. F.)	Viscosity, Saybolt Universal, at 100° F.	Viscosity, Saybolt Universal, at 210° F.	Viscosity Index	Pour Point (Deg. F.)	Color (N.P.A.)	Carbon Residue (Per Cent)	Steam-Emulsifaction No.	Remarks
Run No. 1														
Mid-Continent reduced pressed distillate	....	...	....	25.2	425	485	312	50	66.0	20	4½ dilute	0.3	..	
Primary Product	±81.0	120	65-70	30.4	425	485	227	48	96.0	35	3	0.025	30	
Secondary product	8.5	...	....	19.0	430	480	530	55	25.5	15	8	...	..	
Residual extract	8.0	...	....	11.0	...	...	plus 2,000	76	...	20	black	...	..	
Original stock, after acid and clay treatment	±88.0	...	....	26.0	425	485	288	49	69.0	30	3	0.05	60	10 lb. acid, 5 lb. "filtrol" based on original oil.
Run No. 2														
Mid-Continent neutral distillate	....	...	....	27.5	365	430	128	..	...	25	4½ unstable	0.21	..	
Primary Product	73.0	140	75	31.8	380	430	115	..	...	30	1¾	Trace	60	
Extract	27.0	...	....	16.4	370	440	275	..	...	15	4½ dilute	...	..	
Run No. 3														
Mid-Continent neutral distillate	....	...	....	24.9	420	500	316	..	...	20	7½	0.28	..	
Primary product	75.0	140	60	29.5	430	500	2?2	..	...	30	2¾	0.02	..	
Extract	25.0	...	....	13.1	415	500	1,560	..	...	20	7½ dilute	...	..	
Run No. 4														
Mid-Continent residuum	....	...	....	19.0	520	590	...	305	...	..	black	7	..	7 lb. acid, 29 lb. "filtrol" based on original oil.
Primary product	59.0	200	68	26.5	540	590	...	202	...	50	7½	0.85	...	
First extract	16.5	...	....	...	...	...	...	...	...	..	...	...	..	
Second extract	6.5	...	....	...	...	...	...	...	...	..	...	...	..	
Ceresin (melting point, 160 deg. F.)	8.5	...	....	...	...	...	...	...	...	..	...	...	..	
Original stock after acid and clay treatment	72.0	...	....	24.1	520	590	...	218	...	100+	7½	2.36	..	30 lb. acid, 29 lb. "filtrol" based on original oil.
Run No. 5														
Pennsylvania residuum	....	...	....	24.2	600	670	4,230	2?9	95.5	..	black	...	..	
Primary product	89.5	...	....	26.0	...	...	3,300	205	107.5	..	...	...	..	
Filtered product	....	...	....	...	...	...	...	...	...	..	5½ dilute	...	..	½ lb. acid, 10 per cent "filtrol."
First extract	6.5	...	....	7.5	...	...	...	...	...	..	black	...	..	
Second extract	4.0	...	....	12.4	...	...	...	...	...	..	black	...	..	
Run No. 6														
California distillate	....	...	....	16.8	405	...	1,320	69.8	—34	..	...	...	..	
Primary product	50.0	300	60	23.6	405	...	712	63	43	..	...	...	..	
Extract	50.0	...	....	...	...	...	...	...	...	..	...	...	..	
Run No. 7														
Treated California residuum	....	...	....	19.9	400	...	1,074	76.8	47	..	...	...	..	
Primary product	55.0	275	60	25.1	400	...	726	73	90	..	...	...	..	
Secondary product	31.5	...	....	17.8	...	...	288	46.2	9.5	..	...	...	..	
Extract	13.5	...	....	asphalt, about 100 deg. penetration										
Run No. 8														
California distillate	....	...	....	22.4	...	...	322	50.6	67.1	..	...	...	..	
Primary product	55.0	275	60	28.0	...	...	229	48.2	97.8	..	6	Trace	..	
Extract	45.0	...	....	...	...	...	...	...	...	..	...	...	..	

ages of solvent in order to remove extracts of considerable volume, there will of necessity be a higher proportionate loss of the "paraffinic" material by solution in the solvent phase. All hydrocarbons are somewhat soluble in selective solvents, although varying greatly in degree. To secure a 50-viscosity-index oil from California distillate of from — 30 to — 40 viscosity index, it is necessary to accept a raffinate yield of under 50 percent—using from 250 percent to 350 percent of solvent. To increase further the viscosity index of such raffinate gives even lower yields, which are often so low as to be disproportionate to the gain in tent to 1.6 percent by weight, and doubtless the reduction could have been carried further. On the same sample 35 pounds of 100 per cent acid reduced the sulfur to 2.3 percent, after which point the sulfur content was little affected by further acid—although a total of 100 pounds per barrel was used. The 75 percent yield of raffinate, containing 1.6 percent sulfur was nearly as high as that secured by treatment with 35 pounds of acid (78 percent). The viscosity index was 30 points higher, the color was lighter, the carbon residue reduced, and the resistance to emulsion appreciably improved.

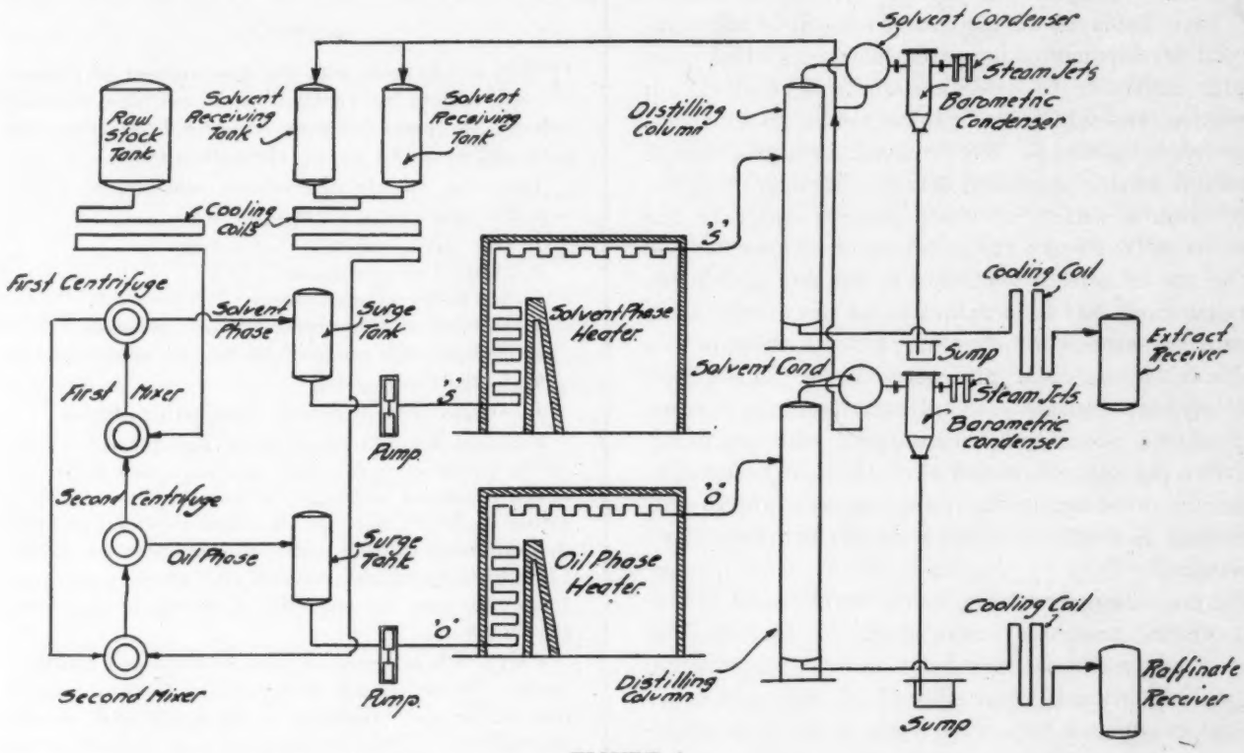

FIGURE 4
Qualitative Flow Sheet of Commercial Solvent-Extraction Process Using Solvent A.

quality. Probably to secure an oil of 100 viscosity index, it will be impossible to secure yields much greater than 20 per cent of the original stock.

Among other types of oil which are found to be materially improved by the solvents and solvent process developed by Foster Wheeler are high-sulfur oils. Such oils will be affected with respect to gravity, flash, viscosity, carbon residue, color, etc., in the same way as would similar oils more or less free from sulfur. In other words, sulfur is a problem quite independent of other characteristics. The effect of proper solvent extraction on high-sulfur oils is drastically to reduce their sulfur content. As an example, consider the treatment of a reduced pressed distillate of about 250 viscosity at 100° F. produced from Mexican Tecomate crude. This distillate contained slightly over 3½ percent of sulfur by weight. An extraction with solvent "A" removed approximately 15 percent as extract, and produced a raffinate of 2.5 percent sulfur; while an extraction removing 25 percent as extract reduced the sulfur con-

FLOW CHART OF PROCESS—AND METHOD OF OPERATION

In order to show briefly the principles which comprise the technology and operation of the Foster Wheeler extraction process a diagrammatic flow sheet for two-phase counter-current flow is shown in Figure 4. Regardless of which solvent, "A," "B," or "C," is used, the flow sheet applies. Between these three solvents the main differences will be solely the temperature to which the two heaters must raise the oil and solvent phases to effect vaporization of the solvent.

In Figure 4 lines marked **O** transport either oil or the oil phase; lines marked **S** transport either solvent or the solvent phase; lines marked **E** transport the extracted portion of oil, freed from solvent.

The operation is as follows: From the storage tanks for raw stock and solvent, these two materials are simultaneously and continuously passed through the tempera-

(Continued on page 452)

Refining of Lubricating Oils *By Solvent Extraction* With Furfural

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ABSTRACT

THIS article deals with the development of a process, employing furfural as a selective refining solvent, for manufacturing superior lubricating oils with well-balanced service characteristics.

The chief factors determining selection of a solvent for lubricating-oil refining are:

1. **Cost and availability of solvent**
2. **Method of application**
3. **Selectivity of the solvent**
4. **Stability and recoverability of the solvent**
5. **Temperature range of application of the solvent**
6. **Toxicity of solvent**
7. **Adaptability to various lubricating stocks.**

Furfural manufactured from agricultural wastes can be produced at low cost, and has a high degree of selective solvent action on a wide variety of lubricating stocks. It is stable in a closed system, and has the advantage of high application temperature (200° F.) which facilitates contact and quick separation from the most viscous oils. Furfural is also non-poisonous.

Furfural is selective on both residual and distillate stocks. Motor testing demonstrates furfural-refined oils to be very resistant to sludging and carbon deposition.

It is estimated that furfural refining costs less than 1.5 cents per gallon. Plant investment will approximate $200 per barrel daily charge capacity, and plant design may employ vessels and equipment present in most refineries.

This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933.

DURING the past few years lubricating-oil refiners have displayed an increasing interest in the commercial development of improved lubricants which offer greater resistance to oxidation, sludging, and carbon deposition, and which are less susceptible to viscosity-temperature variations. The economical manufacture of lubricants having improved service characteristics, derived from a variety of crude sources, has been the objective of extensive research and development.

The use of selective solvents as refining agents for petroleum oils has been found to be one of the most promising methods for effecting the separation of desirable lubricants from lubricating stocks. This article is devoted to a discussion of solvent refining as a means of producing superior lubricating oils, and specifically describes the application and advantages of furfural as a selective refining agent in the manufacture of oils possessing a desirable balance of performance characteristics.

The circumstances leading to the development of solvent-refining processes originate in the fact that no single-type crude oil refined by methods in common use yields lubricants possessing all of those properties desired in superior oils. Frequently it has been necessary to segregate and separately refine special crudes for the production of lubricants meeting special requirements, usually at a considerably increased refining expenditure. On account of the limitations imposed by the conventional refining methods many excellent lubricating-oil crudes heretofore have been rejected for lubricant manufacture. By virtue of the ability more completely to separate undesirable constituents from crude lubricating stocks, solvent refining makes available a much wider source of crude oils for the manufacture of superior lubricating oils.

EARLY WORK ON SOLVENT REFINING

The earliest commercial application of solvent refining was the process developed by L. Edeleanu for the refining of kerosene and lubricating oils with liquid sulfur dioxide.

Much of the early work on the general subject of solvent refining was confined to the fractionation of the light oils into their separate constituents. Aisinman[1] effected a separation of heavy naphtha by means of dissolving in alcohol and fractionally precipitating by the addition of water. Kharitchkoff[2] accomplished a separation by dissolving certain petroleum distillates in amyl alcohol and fractionally precipitating by the addition of ethyl alcohol. M. A. Rakusin[3] in a publication in 1906 described work of Butleroff, carried out as early as 1870, on the cold fractionation by hydrocarbon mixtures by means of solvents. Riche and Halphen,[4] 1894, studied the selective separation of lubricating oils and residua by means of chloroform and alcohol. E. Valenta[5] studied the separation of aromatic constituents from petroleum distillates by means of dimethyl sulfate. Schwartz and Schluter,[6] in 1911, first discussed the solvent-refining of motor oils with acetone. Moore, Egloff and Morrell,[7] in 1918, presented an article on

solubility and separation of various types of hydrocarbons in liquid sulfur dioxide. Egon Eichwald,[8] in U. S. Patent 1,550,523, disclosed the use of furfural for the purification of hydrocarbon oils. Numerous other patents have been taken out from time to time covering use of organic acids, ketones, anhydrides, and alcohols. C. F. Maybery,[9] in studying the heavy fractions of petroleum, proposed the fractionation of hydrocarbons by means of a mixture of ether and ethyl alcohol. H. M. Smith,[10] *U. S. Bureau of Mines Technical Paper No. 477,* describes the fractionation of Cabin Creek crude lubricating fractions by means of acetone, ethyl acetate, secondary butyl alcohol, and nitrobenzene.

RECENT SOLVENT-REFINING DEVELOPMENTS

Recently H. Steinbrecker[11] discussed the solvent fractionation of lignite tar by means of *a-* dichlorhydrin, ethylene chlorhydrin, and benzyl alcohol. Bergstrom, Gikey and Lung[12] determined the solubility of 250 organic compounds in seven of the aliphatic amines at temperatures between 5° and 25° C. Ferris, Birkhimer and Henderson[13] investigated 110 solvents for fractionation of petroleum lubricating oils, and endeavored to correlate selective solvent action with physical properties of solvent. Ferris and Houghton,[14] in 1932, described the process of refining lubricating oils with nitrobenzene. O. P. Cottrell[15] outlined recent developments in the Edeleanu process covering the use of benzol as a modifying solvent with liquid sulfur dioxide. R. K. Stratford, H. H. Moore and S. Pokorny[16] discuss the solvent refining of lubricating oils by means of anhydrous phenol. Page, Buchler and Diggs,[17] of Standard Oil Company (Indiana), presented a recent paper covering the use of β, β'- dichlorethyl ether for the solvent refining of lubricating stocks.

DETERMINING FACTORS IN THE SELECTION OF A REFINING SOLVENT

Numerous solvents and solvent combinations may be employed in the refining of lubricating oils; but the selection of a solvent for refining depends upon many factors, the most important of which include:

1. Cost and availability of the solvents
2. Method of application of solvent
3. Selectivity of the solvent
4. Stability and recoverability of the solvent
5. Temperature range of application of solvent to oil
6. Toxicity of the solvent
7. Adaptability in application to various type stocks.

1. *Cost and Availability of Solvent*—It is apparent that a solvent must be selected which is readily available in large quantities and which can be obtained at a reasonable cost, considering the solvent losses encountered in plant operation.

2. *Method of Application of Solvent*—Five methods of application of the solvent to the oil are possible:

a. Solution of the oil in the solvent followed by separation of the raffinate by cooling or by changing solvent composition by means of a modifying solvent.

b. Batch extraction by mixing, settling, and decanting.

c. Multiple-batch extraction, which employs a fresh portion of solvent in several successive batch extractions.

d. Batch or step counterflow extractions, consisting of passing oil and solvent in counterflow through a number of separate mixers and settlers.

e. Continuous counterflow extraction in a single apparatus.

In refining lubricating oils with solvents it is desirable, if possible, to employ the continuous method of contacting the solvent with an oil; and, in selecting the best solvent for refining, consideration must be given to obtaining intimate contact between the solvent and the most viscous oil and effecting the rapid separation of the extract and refined-oil layers.

3. *Selectivity of the Solvent*—The term selectivity as applied to lubricating oil constituents has been the subject of considerable study and controversy. In the study of selective action of solvents, viscosity index and gravities have been found useful as guides to separation—although viscosity index and gravities alone are not necessarily a criterion of quality. It is generally conceded that the most selective solvent is that solvent which removes the minimum of high-viscosity-index material when effecting a given increase in viscosity index of a lubricating stock. The discussion of the term "selectivity" will be considered hereafter.

4. *Stability and Recoverability of the Solvent*—Obviously, the solvent employed in refining lubricating oils must not react with constituents of the lubricating stock, and must be stable under conditions employed in contacting the solvent with the oil and in the recovery of the solvent from the separate fractions. In general, the solvent employed should have sufficient volatility to be recoverable at a low enough temperature to insure complete removal from the oil fractions without maintaining excessively high operating temperatures in the process.

5. *Temperature of Application of Solvent*—Inasmuch as the solvent-refining process should be capable of handling oils of a wide range of viscosity, it is apparent that those solvents which are applied at elevated temperatures may be expected to possess considerable advantage over those applied at reduced temperatures. High operating temperatures allow the solvent extraction of viscous and waxy oils without the difficulty of low settling rates and poor contact between the solvent and the oil.

6. *Solvent Toxicity*—Another practical consideration in the selection of the solvent is toxicity. Very poisonous solvents must necessarily be employed in completely-

enclosed systems, and adequate provision must be made for protecting the workmen against poisoning by accidental contact with the solvent or its vapors.

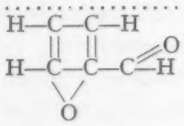
7. Adaptability in Application to Various Type Stocks—A solvent to be employed, generally, in the petroleum industry for the refining of lubricating oils must be capable of application to both residual and distillate oils derived from any type of crude oil.

In selecting a solvent for economically refining a wide variety of lubricating stocks for the production of high-grade lubricating oils, many solvents have been investigated in the laboratories of The Texas Company; and, as a result of the extensive investigations carried out, it has been found that furfural fulfills the requirements outlined above for a superior refining solvent on account of its relatively low cost, its availability in large quantities, high selective action on a wide variety of stocks, stability in solvent-recovery operations, adaptability to counterflow operations, and its non-poisonous properties. Furthermore, furfural lends itself readily to the production of special oils such as high-grade turbine oils, electric-refrigerator oils, white oils, transformer oils, airplane oils, and the like.

1. COST AND AVAILABILITY OF FURFURAL

The properties of furfural are shown in Table 1.

TABLE 1

Structural formula	
Boiling point, °F.	323
Freezing point, °F	—34
Specific gravity at 20°C., sec	1.1622
Viscosity at 100° F., Saybolt Universal, sec.	28.7
Specific heat (68° to 212° F.)	0.416
Heat of vaporization (29.9 in. mercury), B.t.u. per lb	193.5
Vapor pressure at 100° F., mm.	10
Flash point, °F. (Tagliabue closed cup)	138
Solubility in water (96° F.), percent by volume	5.8

Data taken from *Miner Laboratories Bulletin*, "Furfural and Its Derivatives."

The raw material source in the manufacture of furfural is agricultural wastes, such as oat hulls, straw, corn cobs, rice hulls, etc. Its production in commercial practice is comparatively simple, and good yields of solvent are obtained from relatively inexpensive waste materials. Furfural compares favorably in price with practically all of the commercially-available refining solvents.

2. METHOD OF FURFURAL APPLICATION

Figure 1 illustrates the variation of viscosity index with raffinate yields obtained in the furfural refining of an untreated acetone-benzol dewaxed Mid-Continent distillate lubricating stock having a viscosity index, before treatment, of 53. Tests on the charge oil are given in Table 2.

TABLE 2

Gravity, °A.P.I.	21.6
Flash point, °F. (Cleveland open cup)	450
Fire point, °F. (Cleveland open cup)	545
Viscosity at 100°F., Saybolt Universal	1,168
Viscosity at 210°F., Saybolt Universal	79
Pour point, °F. (A.S.T.M.)	—5
Carbon residue, percent	0.87

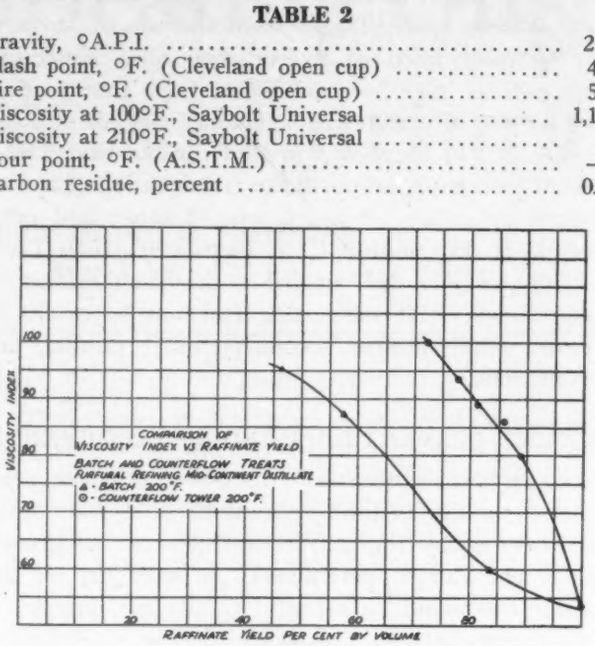


FIGURE 1

Raffinate Yield, Percent by Volume

In Figure 1 the effect of counterflow extraction as compared to batch extractions is clearly shown. The counterflow extractions of this diagram were made in a counterflow tower packed with approximately 12 feet of Raschig rings. All extractions were carried out at temperatures of 200 to 220°F.

Figure 2 illustrates the variation in solvent requirements for obtaining various viscosity indices on the oil

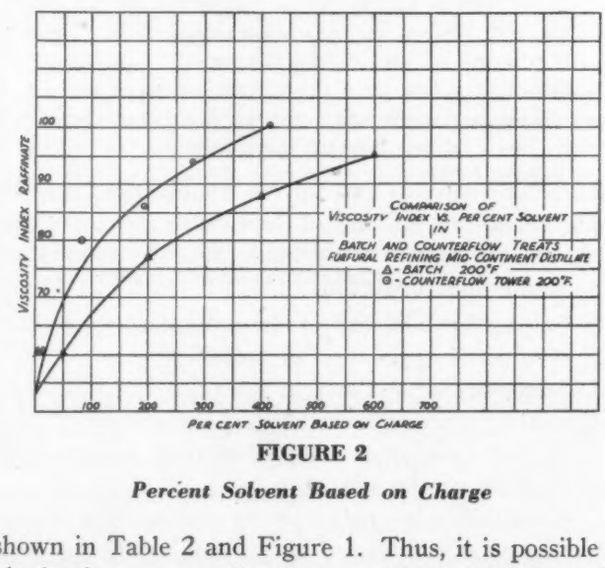


FIGURE 2

Percent Solvent Based on Charge

shown in Table 2 and Figure 1. Thus, it is possible to obtain the same results in counterflow extraction with a little less than one half of the solvent required in batch extractions.

Due to the high temperature of application of furfural to lubricating oils, it is possible to employ continuous countercurrent action in a counterflow tower equipped with suitable provision for distributing the

solvent and oil and means for the prevention of "channeling." At temperatures of 200° to 250°F., where furfural is usually applied, it is possible to process oils of extremely high viscosity, or oils having very high wax content, without involving settling difficulties. The rates of flow have been found to range between 420 gallons and 1680 gallons of charge oil per square foot of tower cross-sectional area per 24 hours, the lower rates being employed on stocks of high viscosity. The usual practice in countercurrent extraction with furfural consists in holding the counterflow apparatus or tower nearly full of oil and maintaining low solvent quantities within the extraction apparatus. This practice greatly reduces the amount of solvent necessary in the refining plant, and tends to keep solvent losses at a minimum.

3. SELECTIVITY OF FURFURAL

Figure 3 illustrates the relation between solvent dosage and extract gravity in furfural refining Mid-Continent distillate. It is noted from Figure 3 that a

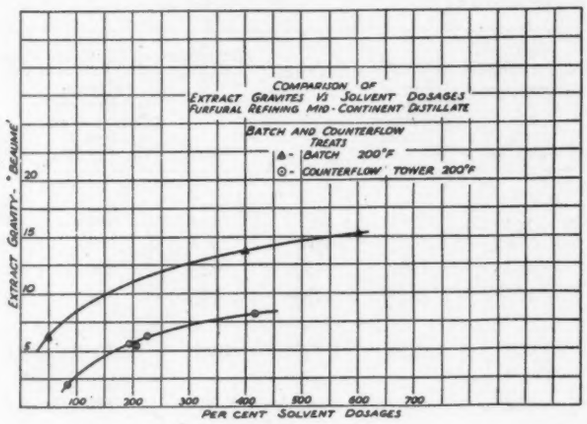


FIGURE 3

Percent Solvent Dosages

much heavier-gravity extract is obtained in counterflow extraction than in batch extraction. The curves of Figure 3 illustrate the high degree of selectivity obtained with furfural and the absence of solvent action on the lighter-gravity constituents.

Figure 4 is presented to illustrate the variation in solvent power of furfural with increasing temperature in batch extraction. Curves are shown for extraction of a different Mid-Continent lubricating stock with varying solvent quantities at 100°F., 200°F., and 250°F. It is believed that the slope of each curve is a relative measure of the selectivity of the solvent under the conditions specified for that curve. It must be remembered, however, that these curves were plotted from the results obtaind in batch extractions; but, as indicated above, much better results would be obtained had counterflow extractions been employed.

It is probable that the solvent begins to lose its selectivity above a temperature somewhere between 225° and 250°F., since the constant-dosage curves in Figure 4 tend to go through a maximum.

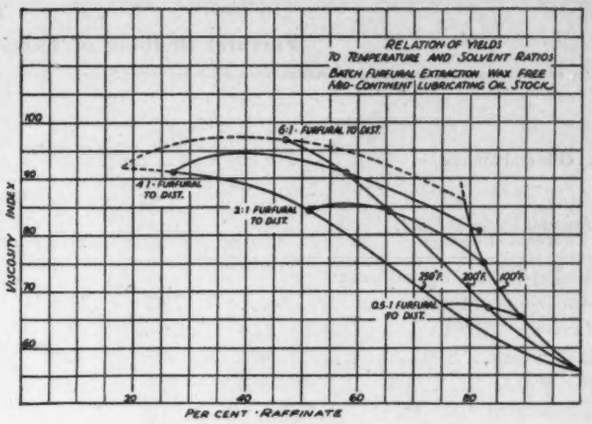


FIGURE 4

Percent Raffinate

4. STABILITY AND RECOVERABILITY OF FURFURAL

In recovering the solvent from the two liquid layers obtained in furfural extraction, two separate solutions are involved, viz., extract solution and refined-oil solution. The composition of these solutions is shown approximately as follows, for an extraction employing 200 percent furfural, by volume:

Extract Solution (Percent)		Refined-Oil Solution (Percent)	
Furfural.....	90	Furfural.....	10
Oil.....	10	Oil.....	90

Recovery of the solvent by vacuum distillation permits operation at vapor temperatures of 210° to 230°F., and at these temperatures it is possible to allow more complete removal of furfural from the oil under moderate operating conditions.

At oil temperatures of 375°F., or above, under 28 inches vacuum, experience has shown that no furfural can be detected in the stripped oils; but provision is made in plant operation to avoid losses in the stripped oils by steaming the remaining traces of furfural from the hot oil, leaving the dry vacuum-stripping unit. Recovery of furfural from the steamings is accomplished by a fractionating system designed after the principles laid down by Mains.[20-21]

Furfural must be kept free from mineral acids, caustic alkali, and ammonia, and should be protected against severe oxidizing conditions. In general, it has been found preferable to recover the solvent under reduced pressure in order to keep solvent quantities in stripped oil at a minimum and in order to prevent polymerization of the extract to a solid or semi-solid condition due to excessive heat. Under reasonably-controlled operating conditions furfural is very stable, and plant losses will not exceed 0.2 percent on the basis of the total solvent used in processing.

5. TEMPERATURE OF APPLICATION

One definite advantage in employing furfural in solvent refining lies in the high temperature of application

TABLE 3

Furfural Refining of Lubricating Stocks from Various Sources

Oils and Source	Lubricating Distillate—Van Zandt Crude				Lubricating Distillate—Pennsylvania Crude				Lubricating Distillate—East Texas Crude		
Amount Furfural Used, Percent of Charge	Charge	Raffinate 200 Percent No Acid	Extract	Dewaxed Refined Oil 5 Lb. Per Bbl. Acid	Charge	Raffinate 300 Percent	Extract	Dewaxed Refined Oil, No Acid	Charge	Raffinate 200 Percent (Batch) Dewaxed and Treated 5 Lb. Per Bbl. Acid	Extract
Yield, percent by volume (solvent refining)	...	78.5	21.5	...	...	76.3	23.3	...	...	75.7‡	24.3
Gravity, °A.P.I.	22.3	26.6	7.6	24.4	29.3	31.0	18.3	29.5	23.7	26.4	8.9
Flash point, °F.	475	495	475	480	395	380	395	420	505	475	470
Fire point, °F.	560	565	565	555	450	425	450	460	575	565	575
Viscosity at 100° F., Saybolt Universal	670	440	14,000*	500	275	270	970	375	910	833	37,000*
Viscosity at 130° F., Saybolt Universal	280	205	2,807	230	142	141	353	180	372	334	6,000*
Viscosity at 210° F., Saybolt Universal	68	60	180	63	53	54	71	58	82	76	274
Pour point, °F.	115	110	60	—10	80	90	70	+10	110	5	65
Carbon residue, percent	0.58	0.18	3.3	0.08	0.57	0.27	3.21	0.33	1.14	0.18	6.35
Viscosity index†	81	95	—163	93.5	110.5	118.5	45	102.5	89	83	....
	waxy	waxy			waxy	waxy			waxy		

Oils and Source	Residuum—Santa Fe Crude			Residuum—Pennsylvania Crude			Residuum—Gulf Coast Crude		
Amount Furfural Used, Percent of Charge	Charge	Raffinate 400 Percent (Batch) Acid-treated 20 Lb. Per Bbl.	Extract	Charge	Raffinate 400 Percent (Batch) No Acid	Extract	Charge	Raffinate 400 Percent (Batch) Acid-treated 20 Lb. Per Bbl.	Extract
Yield, percent by volume (solvent refining)	...	46.8†	53.2	...	76.7	22.5	...	49.7‡	50.3
Gravity, °A.P.I.	17.1	27.5	10.8	27.1	29.3	20.7	17.1	23.1	13.7
Flash point, °F.	405	435	415	465	470	435	425	430	400
Fire point, °F.	470	490	475	530	545	490	495	505	470
Viscosity at 100° F., Saybolt Universal	3,780	460	25,000*	1,025	925	1,300	6,800	2,200*	11,000*
Viscosity at 130° F., Saybolt Universal	1,140	218	4,980*	412	405	485	1,705	749	2,240
Viscosity at 210° F., Saybolt Universal	139	64	297	93	95	90	165	115	170
Pour point, °F.	80	105	95	35	90	70	35	0	40
Carbon residue, percent	8.05	0.72	11.57	1.92	0.97	4.55	3.40	1.02	4.22
Sligh No.	...	0.6	1,256	18.0	0.80	300	...	...	...
Viscosity index†	44	106	—21	99	109	70	+3	64.5	—80
				waxy	waxy				

*Extrapolated viscosity.
†Dean and Davis viscosity index corrected to 0 A.S.T.M. pour test.[20]
‡Acid-treating losses not included in above yields.
All extractions carried out in single-batch operation.
Solvents applied on volume basis.

of this solvent to lubricating stocks. Temperatures between 100° and 250°F. (usually around 200°F.) are employed when furfural is applied to oils. Thus, it is possible to handle extremely viscous or waxy oils with efficient contact between solvent and oil and very rapid settling; and, as pointed out above, this fact alone simplifies the solvent-refining operation and reduces the amount of solvent inventory necessary in any given plant.

Figure 4 illustrates the change of solvent action of furfural on the desirable lubricating constituents with changes in temperatures. In order to obtain fractionation of extract from refined oil it has been found preferable to employ a temperature gradient between the terminals of a counterflow system. For example, in employing 200 percent of solvent, by volume, the furfural would enter the counterflow system at a temperature of approximately 225°F., while the charge oil would enter at the opposite end of the system at a temperature of approximately 100°F. In this manner it is possible to obtain improved fractionation of the more desirable oil from the extract and increase the yield of refined oil to an appreciable extent. Thus, it is possible to utilize the wide temperature range of applicability to a real advantage in the case of furfural.

6. TOXICITY OF FURFURAL

One of the outstanding advantages in the use of furfural is its low toxicity, a very important advantage from the standpoint of operation and repair. In con-

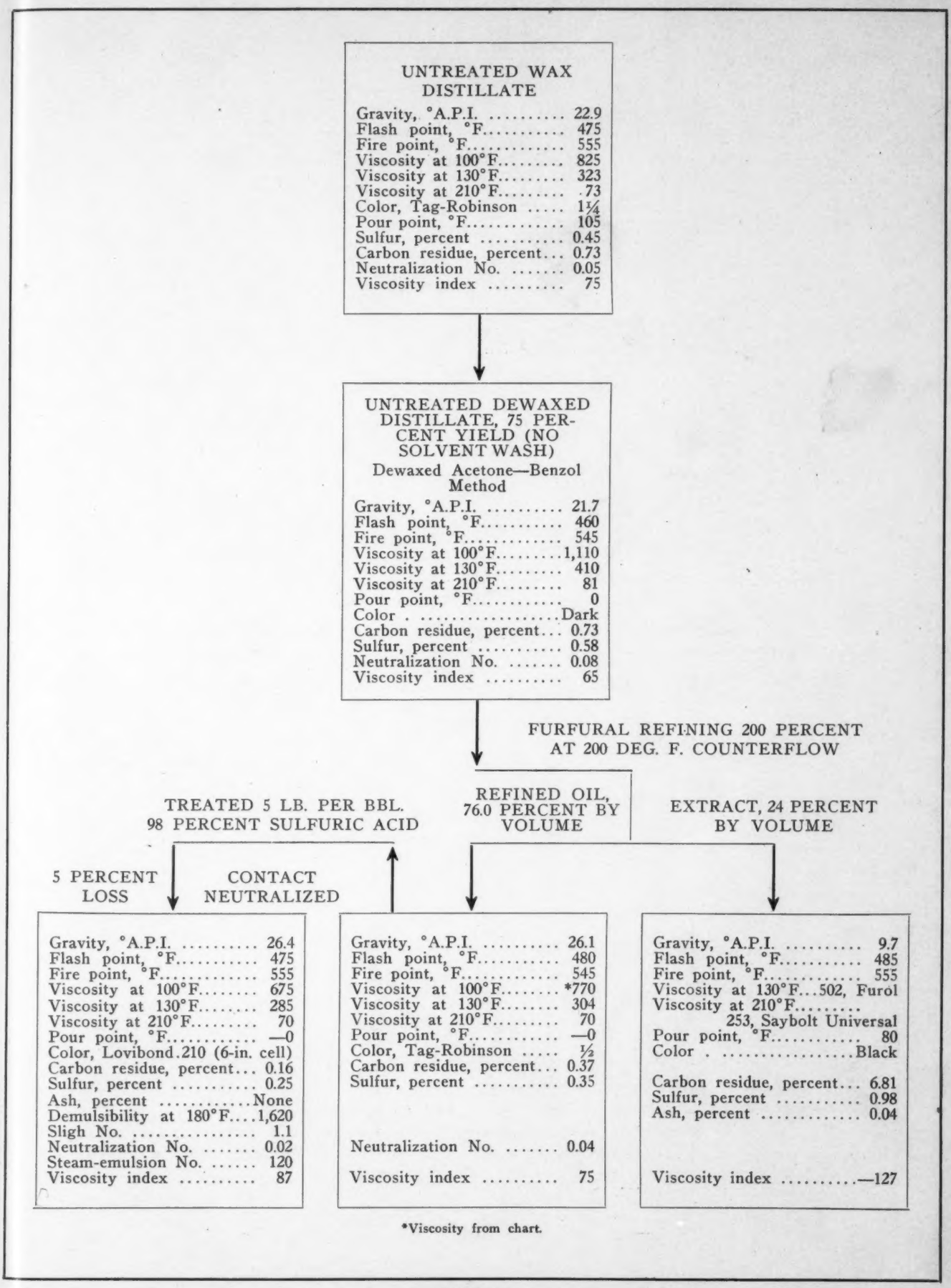

UNTREATED WAX DISTILLATE
Gravity, °A.P.I. 22.9
Flash point, °F..... 475
Fire point, °F..... 555
Viscosity at 100°F..... 825
Viscosity at 130°F..... 323
Viscosity at 210°F..... .73
Color, Tag-Robinson 1¼
Pour point, °F..... 105
Sulfur, percent 0.45
Carbon residue, percent... 0.73
Neutralization No. 0.05
Viscosity index 75
UNTREATED DEWAXED DISTILLATE, 75 PERCENT YIELD (NO SOLVENT WASH)
Dewaxed Acetone—Benzol Method
Gravity, °A.P.I. 21.7
Flash point, °F..... 460
Fire point, °F..... 545
Viscosity at 100°F.....1,110
Viscosity at 130°F..... 410
Viscosity at 210°F..... 81
Pour point, °F..... 0
Color .....Dark
Carbon residue, percent... 0.73
Sulfur, percent 0.58
Neutralization No. 0.08
Viscosity index 65
FURFURAL REFINING 200 PERCENT AT 200 DEG. F. COUNTERFLOW
TREATED 5 LB. PER BBL. 98 PERCENT SULFURIC ACID
REFINED OIL, 76.0 PERCENT BY VOLUME
EXTRACT, 24 PERCENT BY VOLUME
5 PERCENT LOSS
CONTACT NEUTRALIZED
Gravity, °A.P.I. 26.4
Flash point, °F..... 475
Fire point, °F..... 555
Viscosity at 100°F..... 675
Viscosity at 130°F..... 285
Viscosity at 210°F..... 70
Pour point, °F..... —0
Color, Lovibond.210 (6-in. cell)
Carbon residue, percent... 0.16
Sulfur, percent 0.25
Ash, percent .....None
Demulsibility at 180°F....1,620
Sligh No. 1.1
Neutralization No. 0.02
Steam-emulsion No. 120
Viscosity index 87
Gravity, °A.P.I. 26.1
Flash point, °F..... 480
Fire point, °F..... 545
Viscosity at 100°F..... *770
Viscosity at 130°F..... 304
Viscosity at 210°F..... 70
Pour point, °F..... —0
Color, Tag-Robinson ..... ½
Carbon residue, percent... 0.37
Sulfur, percent 0.35
Neutralization No. 0.04
Viscosity index 75
Gravity, °A.P.I. 9.7
Flash point, °F..... 485
Fire point, °F..... 555
Viscosity at 130°F...502, Furol
Viscosity at 210°F.....
253, Saybolt Universal
Pour point, °F..... 80
Color .....Black
Carbon residue, percent... 6.81
Sulfur, percent 0.98
Ash, percent 0.04
Viscosity index .....—127
*Viscosity from chart.

FIGURE 5

Test Flow Diagram for Furfural-Refining Process on Flash-Distilled Paraffinic Lubricating Stock from Mid-Continent Crude, Three-Stage Counterflow

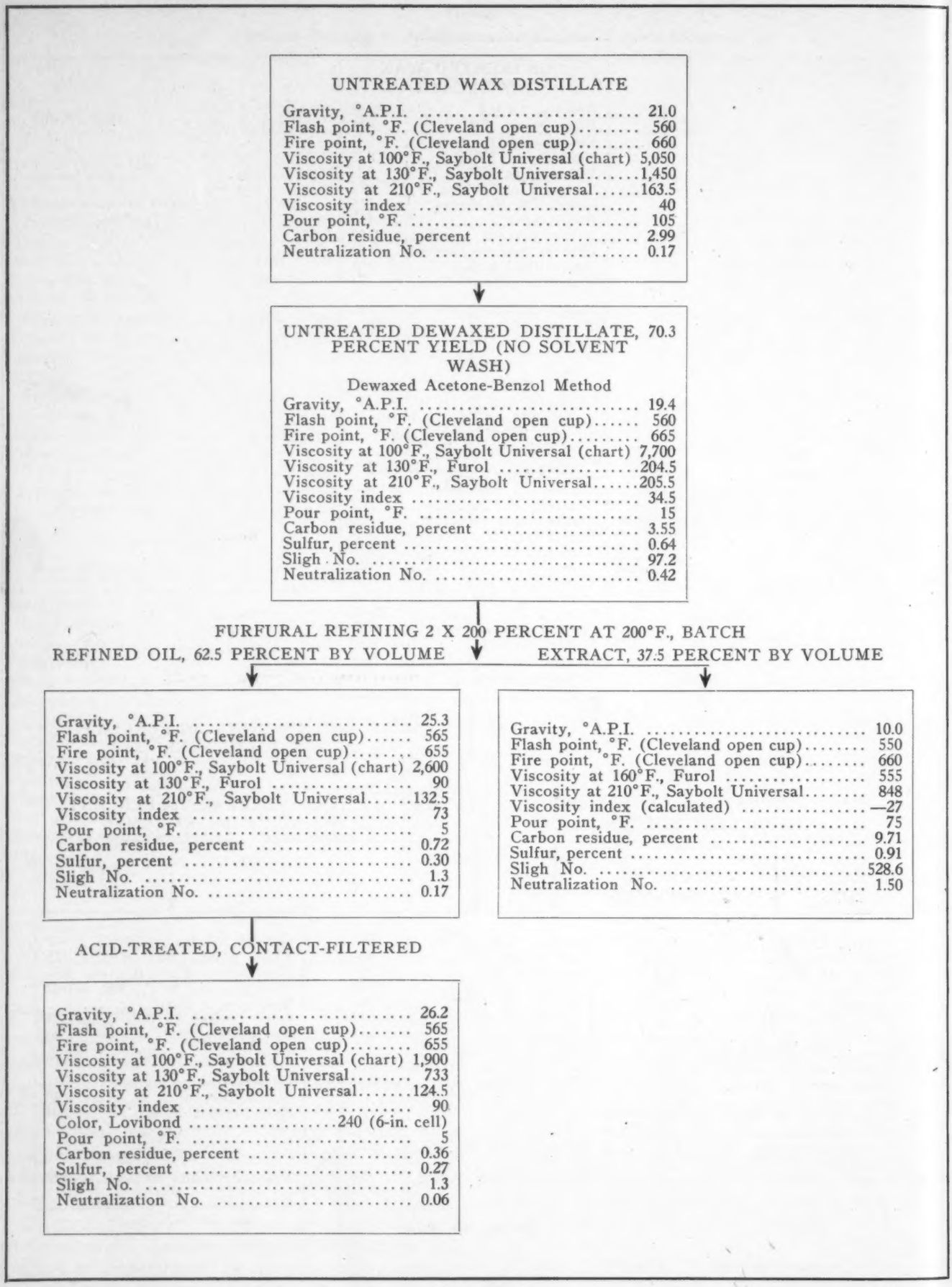

UNTREATED WAX DISTILLATE
Gravity, °A.P.I. 21.0
Flash point, °F. (Cleveland open cup) 560
Fire point, °F. (Cleveland open cup) 660
Viscosity at 100°F., Saybolt Universal (chart) 5,050
Viscosity at 130°F., Saybolt Universal 1,450
Viscosity at 210°F., Saybolt Universal 163.5
Viscosity index 40
Pour point, °F. 105
Carbon residue, percent 2.99
Neutralization No. 0.17
UNTREATED DEWAXED DISTILLATE, 70.3 PERCENT YIELD (NO SOLVENT WASH)
Dewaxed Acetone-Benzol Method
Gravity, °A.P.I. 19.4
Flash point, °F. (Cleveland open cup) 560
Fire point, °F. (Cleveland open cup) 665
Viscosity at 100°F., Saybolt Universal (chart) 7,700
Viscosity at 130°F., Furol 204.5
Viscosity at 210°F., Saybolt Universal 205.5
Viscosity index 34.5
Pour point, °F. 15
Carbon residue, percent 3.55
Sulfur, percent 0.64
Sligh No. 97.2
Neutralization No. 0.42
FURFURAL REFINING 2 X 200 PERCENT AT 200°F., BATCH
REFINED OIL, 62.5 PERCENT BY VOLUME
EXTRACT, 37.5 PERCENT BY VOLUME
Gravity, °A.P.I. 25.3
Flash point, °F. (Cleveland open cup) 565
Fire point, °F. (Cleveland open cup) 655
Viscosity at 100°F., Saybolt Universal (chart) 2,600
Viscosity at 130°F., Furol 90
Viscosity at 210°F., Saybolt Universal 132.5
Viscosity index 73
Pour point, °F. 5
Carbon residue, percent 0.72
Sulfur, percent 0.30
Sligh No. 1.3
Neutralization No. 0.17
Gravity, °A.P.I. 10.0
Flash point, °F. (Cleveland open cup) 550
Fire point, °F. (Cleveland open cup) 660
Viscosity at 160°F., Furol 555
Viscosity at 210°F., Saybolt Universal 848
Viscosity index (calculated) —27
Pour point, °F. 75
Carbon residue, percent 9.71
Sulfur, percent 0.91
Sligh No. 528.6
Neutralization No. 1.50
ACID-TREATED, CONTACT-FILTERED
Gravity, °A.P.I. 26.2
Flash point, °F. (Cleveland open cup) 565
Fire point, °F. (Cleveland open cup) 655
Viscosity at 100°F., Saybolt Universal (chart) 1,900
Viscosity at 130°F., Saybolt Universal 733
Viscosity at 210°F., Saybolt Universal 124.5
Viscosity index 90
Color, Lovibond 240 (6-in. cell)
Pour point, °F. 5
Carbon residue, percent 0.36
Sulfur, percent 0.27
Sligh No. 1.3
Neutralization No. 0.06

FIGURE 6

Test Flow Diagram for Batch Furfural-Refining Flash-Distilled Paraffinic Lubricating Stock from Mid-Continent Crude

tact with the skin it is no more irritating than kerosene, but produces some discoloration. Vapors in moderate concentrations can be inhaled without noticeable after-effect or discomfort. Experience in the manufacture and use of this solvent over a number of years has demonstrated that no ill effects whatsoever are encountered when the solvent is handled with reasonable care.[19]

7. APPLICATION OF FURFURAL REFINING TO A WIDE VARIETY OF STOCKS

Table 3 presents data obtained in the furfural refining of lubricating stocks from a wide variety of crude sources. The extractions in this table are presented only to demonstrate the wide range of applicability of furfural to lubricating stocks. It must be emphasized that these extractions were all carried out in batch; and that had the extractions been carried out in counterflow, much higher yields would have resulted, and the solvent requirements would have been materially reduced. A number of extractions presented in Table 3 were carried out on undewaxed oil, dewaxing being accomplished after the furfural-refining operation. In general, it has been found that dewaxing should precede solvent refining on most lubricating stocks.

The results of Table 3 indicate that furfural is very effective in refining both distillate and residual oils from a very wide variety of sources.

DEWAXING OF PARAFFINIC STOCK PRIOR TO REFINING

The sequence of refining steps has received considerable attention in the development of the furfural-refining process. The conclusions in this respect are that solvent refining generally should be carried out on dewaxed oils, since the solvent-refining operation on raw waxy stocks involves a concentration of the wax in the raffinate portion, and a consequent increased loss of a more valuable oil results in the subsequent dewaxing step. While it is possible that dewaxing may follow solvent refining more economically on some lubricating stocks, it is felt that in most instances this concentration of wax makes solvent refining after dewaxing the preferable and most economical procedure.

It is particularly important in solvent refining that a dewaxing process be employed which completely removes wax, thereby eliminating the possibility of an increase in cold test on account of removal of extract materials.

The acetone-benzol dewaxing process, as described by Govers and Bryant,[18] has been found to be very effective on lubricating distillate stocks from very low viscosities up to viscosities as high as 160 sec. at 210°F., Saybolt Universal. This process has been successful in dewaxing treated residual oils as well as distillate oils.

Figure 5 illustrates the processing of distillate paraffinic lubricating stock from Mid-Continent crude for the production of solvent-refined S.A.E. 40 oil.

Figure 6 illustrates the similar processing of a heavy paraffin distillate from the same type Mid-Continent crude. The extraction carried out in this particular case was by the batch method.

Processing of the two distillates illustrated in Figures 5 and 6 demonstrates the wide range of applicability of both solvent-dewaxing and furfural refining.

ACID TREATMENT OF FURFURAL-REFINED OILS

It is entirely practical and economical to eliminate acid treatment in the furfural refining of distillate lubricating oils. Contact filtration of the solvent-refined distillate oil results in excellent color and cast without the use of any acid whatsoever. However, the employment of a light acid treatment of the order of three to six pounds of acid per barrel, followed by contact filtration, will yield a much better-colored product, and will result in a further improvement in quality and a further increase in viscosity index.

The balance between solvent and acid requirements can be successfully worked out for any given lubricating stock; and this is a problem which each individual refiner must consider in his own manufacturing operations.

Reference to Figures 5 and 6 illustrates clearly the improvement accomplished on solvent-refined oil resulting from a light acid treat.

DESCRIPTION OF FURFURAL-REFINING PLANT

At the present time a plant capable of processing 750 barrels of oil per day is under construction, and it is expected that this plant will be in full operation sometime before November 1.

Plant facilities required for furfural refining of lubricating stocks consist of the following main operating units as shown in the flow diagram (Figure 7).

1. Vertical counterflow extraction tower with preheaters or exchangers for furfural and charge oil together with means for charging solvent and oil at a pre-determined controlled charge rate.
2. Storage for refined-oil solution and extract solution for supplying charge to strippers.
3. Extract-stripping unit for vacuum distillation of furfural from extract solution.
4. Refined-oil-stripping unit for vacuum distillation of furfural from the refined-oil solution.
5. Solvent-recovery system including vapor heat exchanger, condenser, and water cooler, accumulator drum, vacuum pump after cooler and receiver.
6. Two open steam strippers for removal of final traces of furfural from raffinate and extract, respectively.
7. Separating tower and fractionator for recover-

ing furfural from the steamings and drying of furfural prior to its return to the system.

8. Slop solvent and new solvent storage tanks.
9. Storage tanks for extract and refined oil.

OPERATION OF PLANT

The operation of the plant consists in the following steps:

1. Charging untreated oil to the bottom of the extraction tower at a pre-determined temperature, usually 150° to 200°F., together with the pre-determined amount of furfural at a temperature of 200° to 225°F. to the top of the extraction tower below the settling portion of the tower. Allowable charge rates vary between 10 barrels charge oil per square foot of over-all tower area per 24 hours for the high-viscosity oils up to a charge rate of 40 barrels per square foot per 24 hours for the medium- to low-viscosity oils. Levels in the bottom of the extraction tower are controlled by a duo-gravity float controller which regulates the amount of outgoing extract solution and maintains a solution level in the bottom of the extraction tower. Refined-oil solution and extract solution flow from the extraction tower by gravity to surge tanks provided for each of these fractions. Provision is made in each of these surge tanks for removal of any slight accumulation of extract or refined oil carry-over from the extraction.

2. The refined-oil mix is charged at a constant rate to the refined-oil stripping tower through the economizer section of the refined-oil heater, where the temperature is raised to approximately 230° to 250°F. The pre-heated vapor and liquid is then delivered into the refined-oil stripping tower, which is carried under a vacuum of about 28 inches of mercury. The vaporization of the remainder of the solvent in the refined-oil solution is accomplished by direct contact with hot oil being circulated through the refined-oil heater.

The refined oil leaving the tower is flashed into the steaming-out column, where open steam is injected into the oil for the removal of final traces of furfural. This steaming-out tower is carried under atmospheric pressure, the steamings being combined in the furfural-recovery fractionator. The refined oil from the bottom of the steaming-out column is withdrawn and pumped through the interchanger provided for heating the solvent and an after-cooler employing water cooling.

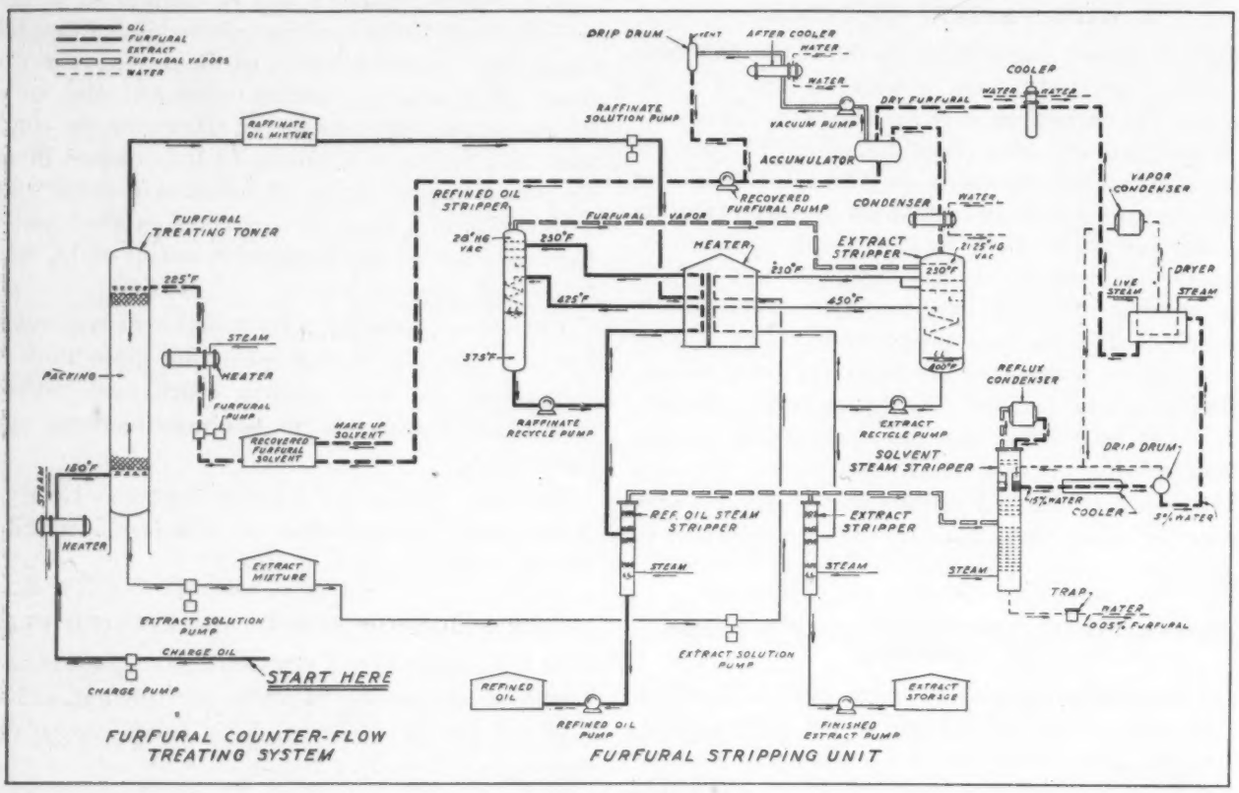


FIGURE 7

3. The extract solution contains approximately 90 percent solvent and 10 percent extract. This solution is pre-heated in the economizer section of the extract heater in the same manner as in the case of the refined-oil stripping system. Approximately 25 percent to 30 percent of the solvent is vaporized in the economizer section, the remainder being vaporized with hot re-circulated extract oil in the extract-stripping tower. Extract accumulation is charged from heater to open-steam furfural-stripping column as in the case of the refined oil; thence it is passed through exchangers to storage.

4. Vapors from the refined-oil stripper tower and extract-stripper tower are combined, and flow through a vapor-heat exchanger, condenser, and the water cooler to the accumulator drum. The vacuum pump takes suc-

tion on the top of the accumulator drum, and the discharge of the vacuum pump is exhausted through an after-cooler for the condensation and recovery of the remaining traces of furfural. The solvent in the accumulator drum may be returned to solvent storage or it may again be employed directly in charging the extraction tower, thereby keeping a small volume of solvent in circulation and affording greater over-all protection to the solvent.

Flue gas, containing less than six percent oxygen, is employed for the protection of the solvent in storage against oxidation.

COST OF INSTALLATION

While it is recognized that each refiner who may consider the installation of solvent-refining equipment may have part of the facilities available for furfural refining, it is estimated that the gross expenditure for the installation of solvent-refining equipment will average approximately $200 per barrel of daily charge capacity.

Much obsolete refinery equipment can be employed in the design of a solvent-refining plant, as no special provision must be made for carrying high pressures and no precautions are necessary to guard against corrosion of equipment on account of solvent decomposition. No refrigeration facilities are required for chilling either oil or solvent or maintaining solutions at a low temperature. Exhaust steam may be used in the plant for maintenance of temperature, and sufficient heat is recoverable from the stripped oils to maintain solvent and oil at the proper operating temperatures.

COST OF FURFURAL-REFINING OPERATIONS

Costs have been estimated for the processing of Mid-Continent distillate lubricating stocks using 200 percent, by volume, of furfural in a counterflow system at 200°F. Obviously, the cost of operation in solvent refining is a function of the amount of oil processed, and the unit cost will be lower in the large installations. The distribution of the costs for the operation of a plant to process 750 barrels per day of a Mid-Continent distillate oil with 200 percent furfural, by volume, is presented in Table 4.

TABLE 4

Operating labor	$0.0527 per 42-gal. bbl.
Operating materials	0.0053
Repairs	0.0130
Fuel, steam, power, and water	0.1210
Pumping and storing	0.0694
Solvent loss (0.2 percent of total solvent used)	0.1575
Depreciation, insurance, and taxes	0.0938
Total	$0.5127

The above figures are considered a very conservative estimate of solvent-refining costs when employing furfural in a plant of the size indicated. It is anticipated that solvent losses may be reduced considerably below the estimate of 0.2 percent by volume. The high figure for fuel, steam, power and water does not allow for heat exchange provided for in the commercial installation.

SOLVENT REFINING FOR VISCOSITY INDICES OF 100 OR ABOVE

Reference to Figures 1 and 2 makes it apparent that a viscosity index of 100 or higher may be obtained in refining Mid-Continent oil by increasing the amount of solvent used, and by sacrificing yield of finished oil. The motor tests presented in this article demonstrate that a furfural-refined oil having a viscosity index of 80 to 90 is a highly satisfactory oil from the standpoint of motor performance; and it is evident that oils in the range of S.A.E. 30 to S.A.E. 60, inclusive, when furfural-refined to a viscosity index of approximately 90, are extremely well-balanced oils with excellent service characteristics, comparing favorably with other premium oils now on the American market.

It is felt that attempts to solvent-refine for viscosity indices in excess of about 90, even on the low-viscosity oils, will result in improvements which are more apparent than real and which are not warranted by the additional cost of refining. In this connection, it must be remembered that for an oil in the range of S.A.E. 10 and 20, an error in the determination of viscosity at 100°F. of one Saybolt second involves an error of one half to one point in viscosity index. An error of one second at 210°F. on the same oil makes a difference of 10 points in viscosity index. Thus, precision of the viscosity determination as carried out in ordinary refinery practice permits only a similar degree of accuracy in the viscosity-index determination.

It is believed, therefore, that solvent refining should aim primarily at quality in the finished product rather than at extremes in the viscosity index.

For example, in counter-current solvent refining with furfural, a Mid-Continent lubricating stock was dewaxed with benzol and acetone and solvent-refined with 400 percent of the solvent, by volume, in a counterflow system. The product showed as in Table 5:

TABLE 5

Gravity, °A.P.I.	28.8
Flash point, °F.	480
Fire point, °F.	560
Viscosity at 100°F., Saybolt Universal	535
Viscosity at 130°F., Saybolt Universal	243
Viscosity at 210°F., Saybolt Universal	66.5
Color, Lovibond	25 (6-in. cell)
Pour point, °F.	0
Carbon residue, percent	0.055
Sulfur, percent	0.19
Sligh No.	1.4
Viscosity index	100

Yields are illustrated in Figure 1.

MANUFACTURE OF SPECIAL OILS

Furfural refining has been found to be especially effective in the refining of oils having special and, more or less, rigid specifications. It is known that furfural

TABLE 6

Tests on Refrigerator Oils and Turbine Oils, Manufactured by Furfural Extraction and Acid Treatment

	Refrigerator Oil from Coastal Distillate	Turbine Oil from Mid-Continent Distillate
Gravity, °A.P.I.	28.5	29.3
Flash point, °F.	385	395
Fire point, °F.	440	450
Viscosity at 100°F., Saybolt Universal	190	192
Viscosity at 210°F., Saybolt Universal	45	46
Color—Lovibond	S.G.	14 (18-in. cell)
Pour point, °F.	—20	0
Carbon residue, percent	0	0.03
Sligh No.	3.5	4.8
Viscosity index	81	94
Sulfur dioxide absorbed at 140°F.	18.3	
Neutralization No.	0.01	0.04
Demulsibility	1,620	1,620
Navy emulsion: Water	...	40-40-0-2 min.
Brine	...	40-40-0-1 min.
Caustic	...	40-40-0-7 min.
Steam-emulsion No.	...	30

refining is an excellent starting point in the manufacture of white oils and other highly-refined products. Similarly, refrigerator oils have been prepared from both paraffinic and Coastal sources which pass the sulfur-dioxide absorption test of less than 20 percent sulfur dioxide, by volume, absorbed at 60°C.

Furfural refining is an excellent starting point for the manufacture of turbine oils. In this case it is particularly important that sludge-forming materials be completely removed in the refining process and that good demulsibility be obtained on the finished product. Table 6 illustrates the tests of turbine oils and refrigerator oils manufactured by solvent refining with furfural.

MECHANICAL TESTING FURFURAL-REFINED OILS

Livingstone and Gruse,[22] 1929, found that the Conradson carbon-residue value on paraffin-base lubricants was indicative of the results to be expected in carbon deposition in poppet-valve motors. Page, Buchler, and Diggs[17] mentioned a parallelism between improvement in viscosity index and oxidation resistance for a given oil upon solvent refining.

Complete mechanical tests have been run on furfural-refined oils and oils refined from the same lubricating stock by usual methods. These tests were carried out under high crankcase temperature conditions to accelerate sludge formation during a relatively short period of operation.

Table 7 is a record of the averages of a large number of mechanical tests conducted to determine the comparative behavior of furfural-refined Mid-Continent oils and lubricating oils now being marketed which are characterized by high viscosity indices. Average tests on the original oils are shown in this tabulation.

HIGH-SPEED AUTOMOTIVE TESTS ON FURFURAL-REFINED OILS

High-speed automotive tests were conducted for the purpose of comparing the behavior of furfural-refined oil with other high-viscosity-index oils.

Six cars were used in this test, and these cars were operated at an average speed of 67 miles per hour for seven hours; after which tests the oils were drained from the crankcase, the crankcase thoroughly washed with solvent, and the sludge separated by centrifuging. The engine after each test was taken down and the carbon deposit scraped off and carefully weighed.

Table 8 presents tests on oils used in this comparison, and presents the comparative motor-oil consumption, fuel consumption, sludge formation, and carbon deposition on the various oils.

It is noted that the furfural-refined oil is superior to all of the other oils from the standpoint of carbon deposit; it is substantially equal from the standpoint of sludge formation. The precision of the mechanical testing is considered to be within approximately five percent on motor-oil consumption figures and probably

TABLE 7

Comparative Tests and Engine Performance of Various Grades High-Viscosity-Index Motor Oils, S.A.E. 40

Speed of engine—900 r.p.m. Oil Temperature—250°F. Jacket Temperature—210°F. Full load, open throttle

	Furfural Refined 200 Percent Counterflow Mid-Continent	Oil No. 1	Oil No. 2	Oil No. 3	Oil No. 4
Oils, S.A.E. 40					
Gravity, °A.P.I.	26.6	27.8	30.7	24.9	28.3
Flash point °F. (Cleveland open cup)	480	465	435	450	465
Fire point, °F. (Cleveland open cup)	560	535	490	505	520
Viscosity at 100° F., Saybolt Universal	675	630	555	640	600
Viscosity at 210°F., Saybolt Universal	69.5	69	67.5	67.5	68.5
Pour point °F.	0	+10	—30	+20	+10
Color, Lovibond	225 (6-in. cell)	4½ (Tag-Robinson)	100 (6-in. cell)	40 (½-in. cell)	180 (6-in. cell)
Sligh No.	2.6	1.7	2.8	3.8	4.4
Viscosity index	85.5	91.5	99.5	83.5	94
Carbon residue, percent	0.19	0.35	0.07	0.41	0.08
Total sludge from crankcase, grams	12.07	11.98	10.94	19.99	11.73
Total carbon deposit, grams	6.14	8.21	7.15	8.91	8.66

TABLE 8

High-Speed Automotive Tests Furfural-refined High Viscosity-Index Lubricating Oils, S.A.E. 50

	Mid-Continent Furfural-refined Oil—200 Percent Counterflow	Oil No. 5	Oil No. 6
Gravity, °A.P.I.	26.1	28.2	27.8
Flash point, °F.	515	460	455
Fire point, °F.	600	520	530
Viscosity at 100°F., Saybolt Universal	1,002	649	654
Viscosity at 130°F., Saybolt Universal	395	286	285
Viscosity at 210°F., Saybolt Universal	82	74	73
Pour point, °F	—5	+20	+15
Color, Lovibond	50 (½-in. cell)	¼ (Tag-Robinson)	¼ (Tag-Robinson)
Carbon residue, percent	0.25	0.83	0.73
Sligh No.	1.6	4.3	4.4
Viscosity index	80	+100	99
MOTOR TESTS			
Motor oil, miles per gallon	365	354	328
Fuel, miles per gallon	11.9	11.9	11.9
Speed average	67	67	67
Duration, hours	7	7	7
Sludge, grams	15.1	15.7	16.8
Carbon deposit, grams	8.9	12.9	13.1
No. of cars	6	6	6

more accurate in the carbon and sludge determination.

Attention is called to the fact that the furfural-refined Mid-Continent oil was refined only to a viscosity index of 80. This drastic motor test does demonstrate, however, that the furfural-refined oil is a very well-balanced oil in all of its performance characteristics, and gives further evidence that viscosity index is not the sole criterion of quality.

CONCLUSIONS

After a prolonged investigation involving numerous solvents, furfural has been selected as meeting the requirements of the most desirable selective refining solvent.

The factors which determine the selection of furfural as a refining solvent for lubricating oils are as follows:

1. Furfural is manufactured from agricultural wastes by a relatively simple process, and is available in large quantities at a reasonable cost.

2. Furfural can be applied even to the most viscous or waxy oils in a single counterflow tower due to its high temperature of application of 200° to 250°F. Furfural is extremely selective, and has a very low solubility for the desirable components of a lubricating stock—resulting in high yields of refined oil.

3. Furfural does not react with the oil under the conditions encountered in the extraction or solvent-recovery operations.

4. A greater range of temperature of application is possible with furfural than with most solvents.

5. Furfural is non-toxic, and requires no more handling precautions than does kerosene.

6. Furfural has been demonstrated to be adaptable to both distillate and residual oils from Mid-Continent, Coastal, Pennsylvania, and California crudes.

By confirmatory mechanical tests furfural-refined oils have been demonstrated to be well-balanced high-grade lubricating oils capable of resisting oxidation, sludge formation, carbon deposition, and extreme viscosity-temperature variations. Furfural refining as an auxiliary step in the manufacture of special oils such as turbine oils, transformer oils, electric-refrigerator oils, airplane oils, white oils, and the like, has demonstrated its high degree of effectiveness.

Cost of a furfural-refining plant will average approximately $200 per 42-gallon-barrel daily capacity. Cost of solvent-refining operations, including fixed and overhead charges, will approximate $0.51 per 42-gallon barrel of charge oil.

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Scope and Flexibility of the Edeleanu Process . . .

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This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933

SOLVENT refining pioneered by L. Edeleanu, the inventor of the liquid-sulfur-dioxide process, has become the recognized method of refining for practically all types of petroleum fractions. Solvent refining is not a new development in the petroleum industry. For the past decade the Edeleanu process of solvent treating of kerosenes with liquid sulfur dioxide has been recognized and acknowledged by the oil industry as being the most successful and efficient method of production of high-grade kerosenes. The use of this process is world-wide in scope; and in spite of the many advancements and improvements in recent years in refinery practices and methods of processing, the Edeleanu solvent treatment of kerosenes stands foremost in the manufacture of the highest quality kerosene.

The scope of the process has been steadily increased over the last several years, until at the present time many Edeleanu plants throughout the world are operating not only for the treatment of kerosenes, but also for the treatment of naphthas and all types of lubricating oils.

SOLVENTS-REFINING ART WIDENED

The art of solvent refining was such a drastic departure from the old-established method of acid treatment that many refiners were slow to accept the value of this new type of refining for treating lubricating oils. However, the increasing demand over the last few years for better quality—which was caused chiefly by new engine design and ecomonical refinery operation—has brought about tremendous activity in the solvent-refining field which has resulted in the development of several new solvent-refining processes. Just as during the last few years all refiners have been directing the major part of their activities toward the manufacture of high-octane gasoline, so today their attention is directed largely upon the manufacture of solvent-refined oils.

There are many different compounds that are oil solvents to some extent, and are so classified, yet which would be impractical for use in commercial refining. Some of these compounds have too low a solvent or extractive power; others have too low a degree of selectivity; some are not stable; others injure the oil; some are suitable only for certain types of oil; while others are difficult to recover, or else build up undesirable additional products.

A year ago, at the Thirteenth Annual Meeting of the American Petroleum Institute, at Houston, Texas, the author presented a paper outlining the activities and progress of The Edeleanu Company in the sphere of solvent refining pertaining to the production of motor oils of improved specification. Data were submitted indicating the results that were obtained by the new developments with the Edeleanu process, viz., the treatment of lubricating oils with a mixture of liquid sulfur dioxide and benzol. Since that time much experimentation has been made, further confirming the value of this process for the treatment of lubricating oils.

SOLVENT POWER INCREASED

One of the very important features of this new development is the ability to increase the degree of solvent power of liquid sulfur dioxide by the addition of varying quantities of benzol. By this innovation the most extreme and diversified types of oils of a wide range of gravities and viscosities can be satisfactorily refined by using different proportions of the two solvents to satisfy the requirements both as to high quality and high yields.

The benzol — sulfur-dioxide process is unique in that it utilizes a combination of two solvents—both having extraordinary solution power for unsaturated, aromatic, resinous, sulfur-containing, and otherwise non-paraffinic compounds. On account of this discovery of the blending of benzol and sulfur dioxide in varying proportions, the Edeleanu process stands out as one having great scope and flexibility.

Changes in design and construction of Edeleanu plants now make possible the treatment of gasolines or kerosenes with sulfur dioxide, and lubricating oils with sulfur dioxide and benzol—sulfur dioxide in the same unit—thereby effecting considerable economy in equipment cost by utilizing the remarkable adapt-

ability of the process in the refining of the light, intermediate, and heavy products of distillation.

The wide range from transformer and neutral oils to cylinder and bottom stocks requires, for utmost efficiency, both solvent and selective power that can be regulated to meet the varying requirements of these different viscosity oils of naphthenic and paraffinic sources. Lighter oils normally require a more selective-solvent action than heavier oils to obtain results consistent with economical plant operation regarding yield; and with the two solvents, benzol and sulfur dioxide, the selectivity—as well as the solvent power—can be governed by different proportions of the two solvents.

CAPITAL INVESTMENT IMPORTANT

The question of capital investment is one of great importance, particularly when the life of the process is considered. Therefore, with the combination of sulfur dioxide or benzol—sulfur dioxide in the same unit, the chance of the process becoming obsolete is lessened considerably. With conditions changing so rapidly in the industry, the refiner never knows what his requirements will be a year hence; and, therefore, in selecting a process, serious consideration should be given to its flexibility and the range of products that can be satisfactorily treated.

Due to the selective solvent power of sulfur dioxide alone, all low-boiling fractions can be very satisfactorily separated into their component parts. The treatment of kerosenes is well known and recognized; but the treatment of lower-boiling fractions, such as naphthas, for the production of special solvents and anti-knock compounds is a field for future development. On account of the effective manner in which sulfur dioxide can refine naphthas at a low cost of operation, it is not unlikely to expect that, with changes in the price structure of gasolines, the sulfur-dioxide process will play an important part in the refining of various gasolines, both heavy and light.

PROCESS FLEXIBLE FOR LUBRICANT REFINING

For the production of high-grade lubricants, with large yields and resulting low costs, the treatment with sulfur dioxide alone is very satisfactory, and is being used extensively for this purpose at the present time. For production of oils meeting certain specifications — including high viscosity index, low carbon residue, improved stability, etc.—the combination of benzol—sulfur dioxide fills the requirements of a very flexible type of operation.

Distillates from various types of crudes throughout the country require a certain treatment to produce the maximum yield of the desired oil at the lowest cost of production. On account of the flexibility of the benzol — sulfur-dioxide combination, these various types of distillates can be refined successfully by varying the proportion of benzol to sulfur dioxide, and also by varying the total volume of solvent. In addition to the variations in quantity and ratio of solvents, the temperature may also be varied to meet certain conditions for treating only lubricating oils. It has been found that lubricating distillates can be refined very successfully at temperatures ranging from 80 to 100°F., without serious reduction in yield of finished oil.

PROCESS GOOD FOR HIGH VISCOSITY-INDEX OILS

In addition to the wide range of flexibility for treating all types of distillates, from the lightest to the heaviest, with sulfur dioxide or with benzol—sulfur dioxide, there is another outstanding advantage of the process which is particularly adapted to the refining of oils when the yield of oil of high viscosity index is rather low. For example, a distillate from Kettleman Hills crude in California is first treated with sulfur dioxide alone, with the production of approximately 70 to 75 percent raffinate. This raffinate is then further treated with a mixture of benzol and sulfur dioxide. The raffinate from this treatment represents a 90 + viscosity-index oil of very high quality; and the benzol—sulfur-dioxide extract is an oil with properties satisfactorily meeting the requirements of a second-grade motor oil or industrial lubricant. On account of this type of operation the cost of producing 90 viscosity-index oil is greatly reduced. Table 1 indicates the results of this type of operation on distillate and long residuum.

TABLE 1
Results of Test with Sulfur Dioxide Followed by Benzol and Sulfur Dioxide

	Kettleman Hills Dewaxed Distillate (Stock extracted with 150 percent by volume of sulfur dioxide, followed by 200 percent by volume of benzol and sulfur dioxide (30:70))				Kettleman Hills Long Residuum (Propane-treated stock extracted with 200 percent by volume of sulfur dioxide at 115°F., followed by 250 percent by volume of benzol and sulfur dioxide (30:70) at 90°F.)			
	Dewaxed Distillate	Benzol-Sulfur-Dioxide Raffinate	Benzol-Sulfur-Dioxide Extract	Sulfur-Dioxide Extract	Propane-Treated Residue	Benzol-Sulfur-Dioxide Raffinate	Benzol-Sulfur-Dioxide Extract	Sulfur-Dioxide Extract
Yield, Percent by Volume.. ...		35	36	29	..	31.6	44.4	24.0
Gravity, deg. A. P. I.....	18.0	28.0	21.0	6.0	19.1	26.2	19.2	9.9
Viscosity at 210 deg. F., Saybolt Universal	87	66	85	150	98	88	97	131
Viscosity index	0	90	30	..	30	90	39	..

The present Edeleanu plant has been designed to take full advantage of the flexibility of the process. Either treatment of light or heavy products with sulfur dioxide alone, or the treatment of heavy products with benzol—sulfur dioxide of varying proportions, can be carried out in the same unit at low operating costs. The unit is sufficiently flexible to permit the use of the plant for the refining of low-boiling compounds and lubricating oils requiring only sulfur dioxide; and, without serious delay and loss of production, the unit can be converted to the treatment of the higher-boiling hydrocarbons with benzol—sulfur dioxide.

Liquid sulfur dioxide and benzol are two well-known stable products, and can be used over and over in the system without any decomposition. The two solvents are commercially available at rather low costs throughout the country.

A flow diagram of a plant for treating with sulfur dioxide or with benzol—sulfur dioxide is shown in Figure 1.

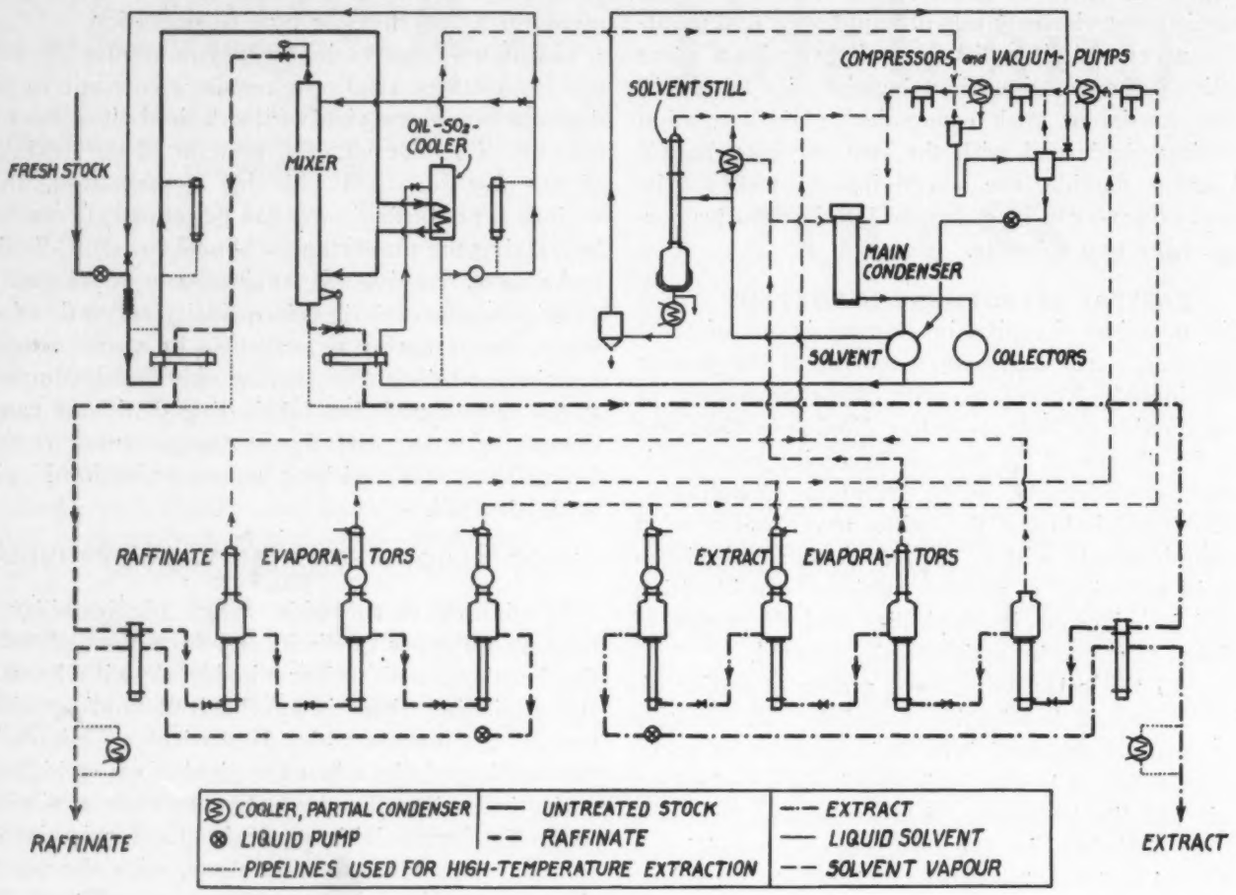

FIGURE 1
Sulphur Dioxide or Benzol — Sulphur-Dioxide Treatment of Hydrocarbons.

PROCESS ADVANTAGEOUS FOR HYDROCARBONS

Solvent refining seems to be approaching the ideal method of processing various types of hydrocarbons, and is being looked upon very favorably by many refiners. Many oil companies are seriously considering the installation of solvent-refining plants for the purpose both of improving the quality of their oils and of reducing the cost of manufacture, and for smoother refinery operation. In choosing a process the refiner naturally desires to have the maximum flexibility commensurate with economical operation.

The Nitrobenzene Process...
Installation and Operating Costs

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THIS paper discusses the installation and operating costs of the nitrobenzene process from the point of view of its application to a number of charge stocks covering a wide range both of viscosity and crude source. An approximate estimate is given of a plant to process 1000 barrels (50 gallons) per day of charge, with all service requirements stated in some detail. Data obtained from experiments on 28 stocks from light distillate to residuums, and derived from crudes varying in type from Pennsylvania to Gulf Coast, are correlated to show the general relation of the viscosity-gravity constant of the stock to the yield of nitraffins† of varying degrees of paraffinicity. Operating costs are given, and variation of cost with variation of solvent-oil ratio; finally, the yield and cost data are combined in a family of curves showing the variation of cost with quality of stock and product. The susceptibility of the product to dewaxing and decolorizing processes is discussed.

This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Ill., October 25, 1933.

THE interest in the use of selective solvents as a means of obtaining paraffinic Pennsylvania-like lubricating oils from all crudes is sufficiently widespread to indicate the probability that the method will be widely accepted as a conventional step in the process of refining crude petroleum. The magnitude of the investment called for by new equipment and the extent to which the new process will change present manufacturing costs are, therefore, timely subjects of discussion.

Intensive work in extraction processes is quite recent, and the effort of each refiner participating has been largely directed towards adaptation of the process to particular stocks, products, and manufacturing conditions. In view of this pre-occupation and of the great variety of crude sources, of stocks from each crude, and of the variable condition of each solvent application, figures generally applicable and comparable are difficult to obtain. So far as nitrobenzene is concerned, the figures given hereafter are not intended to reflect the exact cost for any one set of conditions, but to give the general order of magnitude of costs over a wide field.

The process is essentially simple: The charge oil is mixed with the requisite quantity of nitrobenzene, chilled, allowed to settle, forms two layers—the lower containing the naphthenics in solution in the bulk of the nitrobenzene, the upper the solution of paraffinics; each layer is separated continuously, passed over steam coils in a vacuum evaporator in which all but one percent to two percent of the nitrobenzene is removed, and thence through a vacuum stripping tower, where the remainder is removed by the application of live steam. The general arrangement of the apparatus and the flow of materials and heat are shown in Figure 1.

The chillers for the charge are of the scraper type to permit handling wax-bearing and highly-viscous charges.

The five-stage counter-current extractor is a conventional series of settling and mixing tanks, with the exception that the mixer before the first stage is omitted, the charge being mixed with second-stage nitrene‡ in the chiller; this in order to decrease the wax concentration and improve heat transfer.

The two evaporators are of the cascade type, consisting of closed steam coils placed in a vertical series of shallow trays; each evaporator is split into several units, each unit communicating throughout its length with a corresponding condensing unit.

Pressures of 30 mm. mercury in the first several units and 10 mm. in the remaining units are maintained by means of a three-stage steam-jet vacuum pump. The temperature through the evaporators will vary from 215° F., at the top, to 325° F., at the exit; the temperature of both streams is raised to 350° F., by means of a separate heater before entering the vacuum steam strippers. These operate at a pressure of 50 mm. mercury, in order to permit condensing the steam with ordinary cooling water. To avoid loss of nitrobenzene in the air discharged by the vacuum pumps, the inter-stage cooling is done by surface condensers. The nitrobenzene is recovered by distilling the condenser water under 100 mm. pressure, removing about 10 percent. The remaining 90 percent is practically free of nitrobenzene; the 10 percent

†Solution of the paraffinic fraction in nitrobenzene.
‡Solution of the naphthenic fraction in nitrobenzene.

overhead separates into two layers: recovered nitrobenzene and nitrobenzene-saturated water; the latter is re-circulated to the still. (Note: This recovery still is not shown in the diagram, but is included in the cost.) Recovery of 95 percent of the dissolved nitrobenzene is obtained.

Since nitrobenzene boils at 411.5° F., at 700 mm., and mixtures with oil must be held below 350° F., to prevent decomposition, distillation at low pressures is inherent in the process.

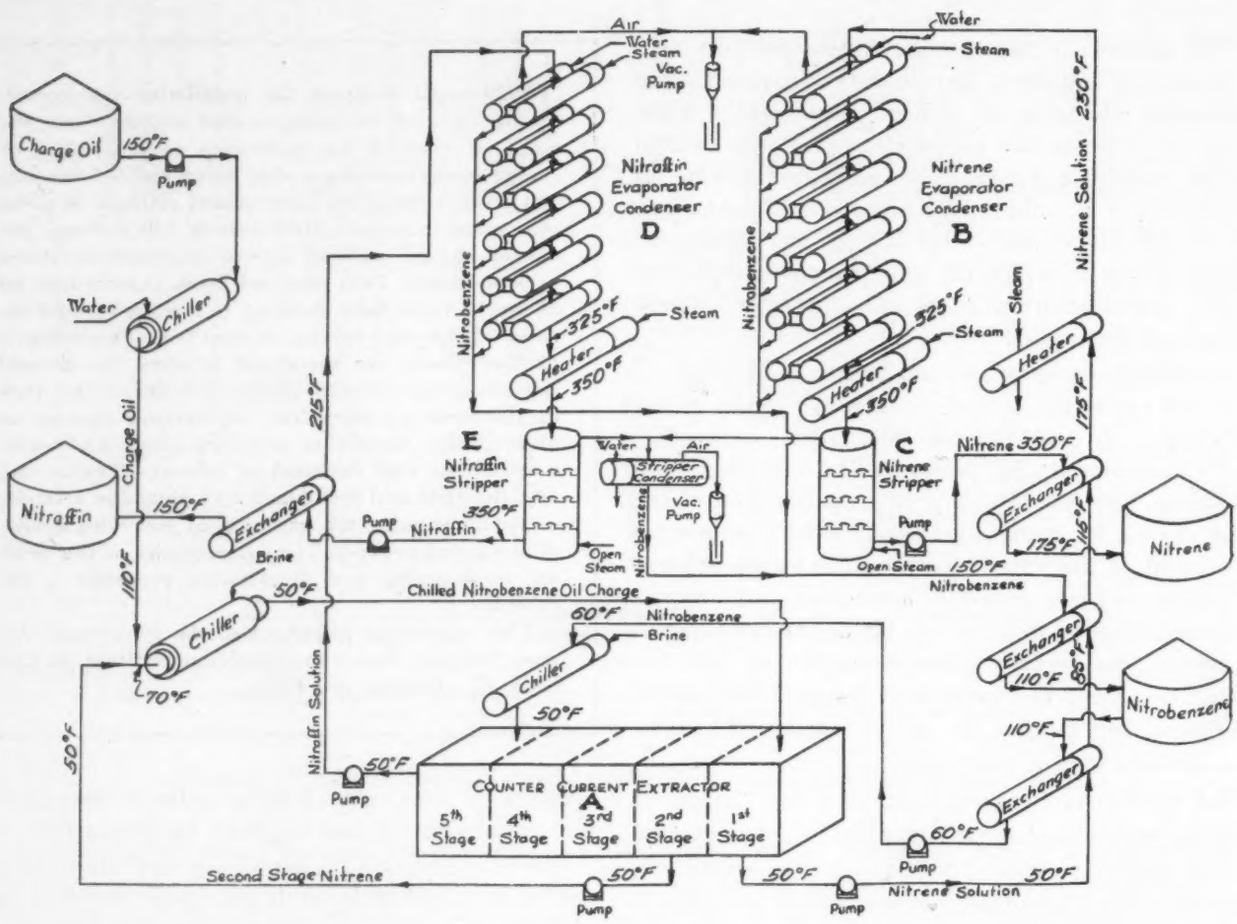

FIGURE 1

INSTALLATION COSTS

Nothing more is attempted than to indicate the general order of the installation cost for a plant to handle 1,000 (50-gallon) barrels of oil per day, under conditions which appear reasonably representative and flexible. Table 1 shows the approximation in detail of the cost of such a plant, capable of recovering 1,500 barrels of nitrobenzene per day, permitting a variation in nitraffin yield of from 40 percent to 60 percent without affecting the charge rate, and independent of the character of the stock—distillate or residual. The total cost is of the order of $222,000 without overhead charges for the apparatus, and $42,500 for the initial quantity of solvent required. A plant to handle only distillate stocks would cost less and require less initial solvent; otherwise a plant of this size, designed for a specific stock, would cost more or less as the ratio of solvent to stock increased or decreased.

OPERATION COSTS

The size of the nitraffin yield becomes increasingly important as a cost factor as the unit value of the nitrene relative to that of the charge stock decreases. If the stock is produced incidental to other operations in larger quantities than required, its value may be that of fuel oil or cracking stock and the same as that of the nitrene produced. In this case the sole cost of raw material accruing to the nitraffin will be the volume of the latter times the unit value of the charge; and the cost per unit of nitraffin will, therefore, be independent of yield. If suitable charge stocks for the process are not available, any costs incidental to added distillation, preliminary dewaxing, or pre-treatment of any kind are all proportionately magnified as the nitraffin yield decreases. The same thing is true if for any reason the unit value of the nitrene is less than that of the charge stock.

The conditions which govern the cost of preparing the stock are too specific and variable to permit of further generalization here beyond the qualitative statements just made. Large nitraffin yields also favorably affect installation and operating costs, but

here the volume of solvent per volume of solaffin* becomes a major factor.

In Tables 2 and 3 are given the results of nitrobenzene extraction on 28 different stocks, roughly classified as of "low" and "high" wax content as the pour points were below or above 75° F. The stocks vary from light lubricating (266 sec., Saybolt Universal, at 100° F.), through overhead motor and cylinder stocks, to residuals of varying asphalt content; and originate in a diversity of crudes from Pennsylvania, through Mid-Continent, to extreme Gulf Coast. This wide variation is illustrative of the flexibility of the process in this respect.

Although these individual experiments were made independently over a long period of time under somewhat haphazard conditions as to temperatures and solvent-oil ratios, the attempt to show a definite relationship between the viscosity-gravity constant of the stock and the yield of nitraffins, and the variation in nitraffin yield with quality, was reasonably successful. Firgure 2 is presented as typical of the correlation of the data on variation of nitraffin yield with quality; Figure 3 of variation of nitraffin yield of given quality with the variation of viscosity-gravity constant of the stock. It will be noted that in drawing the curves in Figure 3 the points resulting from experiments in the waxy stocks were neglected, with rather surprisingly smooth correlation of the remainder; also that the higher wax-content stocks gave consistently higher yields, as might be expected.

*Solution of the paraffinic fraction in solvents generally.

TABLE 1
Nitrobenzene Plant

Capacity:
Charge 1000—50-gal. bbl. of distillate or residual oil per day
Charge 1500—50-gal. bbl. of nitrobenzene per day
To process:
A maximum of 600 bbl. per day of nitraffin
A maximum of 600 bbl. per day of nitrene
A maximum of 860 bbl. per day of nitraffin solution
A maximum of 1,790 bbl. per day of nitrene solution

Assumed conditions:
Charge oil delivered to plant at 150° F.
Extractor operated at 50° F.
Steam at 150 lb. per sq. in. gage available
Fresh water at maximum summer temperature of 85° F. available

Approximate cost of plant (battery-limit lines, charge-oil and product-oil tanks not included):
Exclusive of overheads on material and labor

1. Refrigeration plant, including housing 65 tons at $400 per ton (45 tons delivered to oil and nitrobenzene)	$ 26,000
2. Scraper-type chillers—2500 sq. ft. at $6.00 per sq. ft.	15,000
3. Extractor, five—13 ft. x 5 ft. tanks; four—3½ ft. x 3½ ft. tanks, 4 turbo mixers	10,000
4. Tubular equipment—11,000 sq. ft. at $2.75 per sq. ft.	30,000
5. Evaporator condenser units—5,500 sq. ft. at $5.00 per sq. ft.	28,000
6. Stripper towers, two—3 ft. x 30 ft.; 10 plates	7,000
7. Pumps, eight 5-hp. motor-driven; ten 1.5-hp. steam-driven; three steam-jet vacuum pumps	6,000
8. Nitrobenzene tank and receivers	7,000
9. Nitrobenzene, one recovery evaporator (recovery of nitrobenzene from water)	3,000
10. Control and pump house	4,000
11. Piping (battery-limit lines only)	25,000
12. Instruments	20,000
13. Miscellaneous (paving, drainage, wiring, sanitation, fire protection)	11,000
14. Tools and contingencies	10,000
15. Engineering	20,000
Total	$222,000
Initial charge of solvent—1,000 bbl. (50-gal.) at $0.85 per lb.	$ 42,500

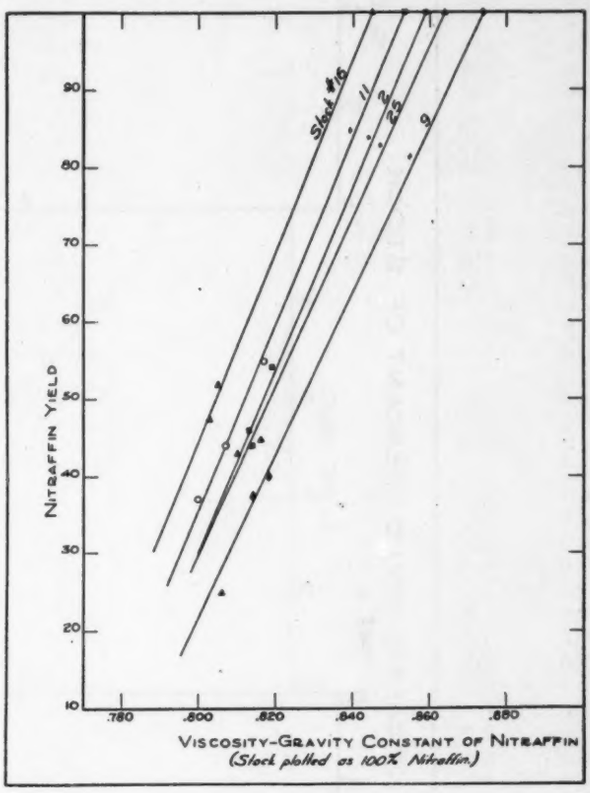

FIGURE 2

One other marked deviation occurs in the case of the Winkler residuals, where the actual yields are 10 percent to 15 percent higher than indicated by the smooth curve. Here the viscosity-gravity constant is distorted by the relatively high asphalt content—the constant for the actual oil of the residual being about 0.015 lower than that of the residual itself.

From data of the character illustrated in Figure 2 and 3, the set of smooth curves of Figure 4 were constructed—showing the general relationship between yield of nitraffin for low-wax stocks varying from 0.800 to 0.900 viscosity-gravity constant, and for nine nitraffins varying in quality from 0.790 to 0.830 viscosity-gravity constant.

DIRECT MANUFACTURING COSTS

At this time consumption of power, labor, and materials other than charging stock can be approximated only by estimate, since no figures from actual operation are available. All quantities are stated in the accompanying tables, and to them rounded unit costs have been applied in an effort to present the general order of magnitude of the total and the rel-

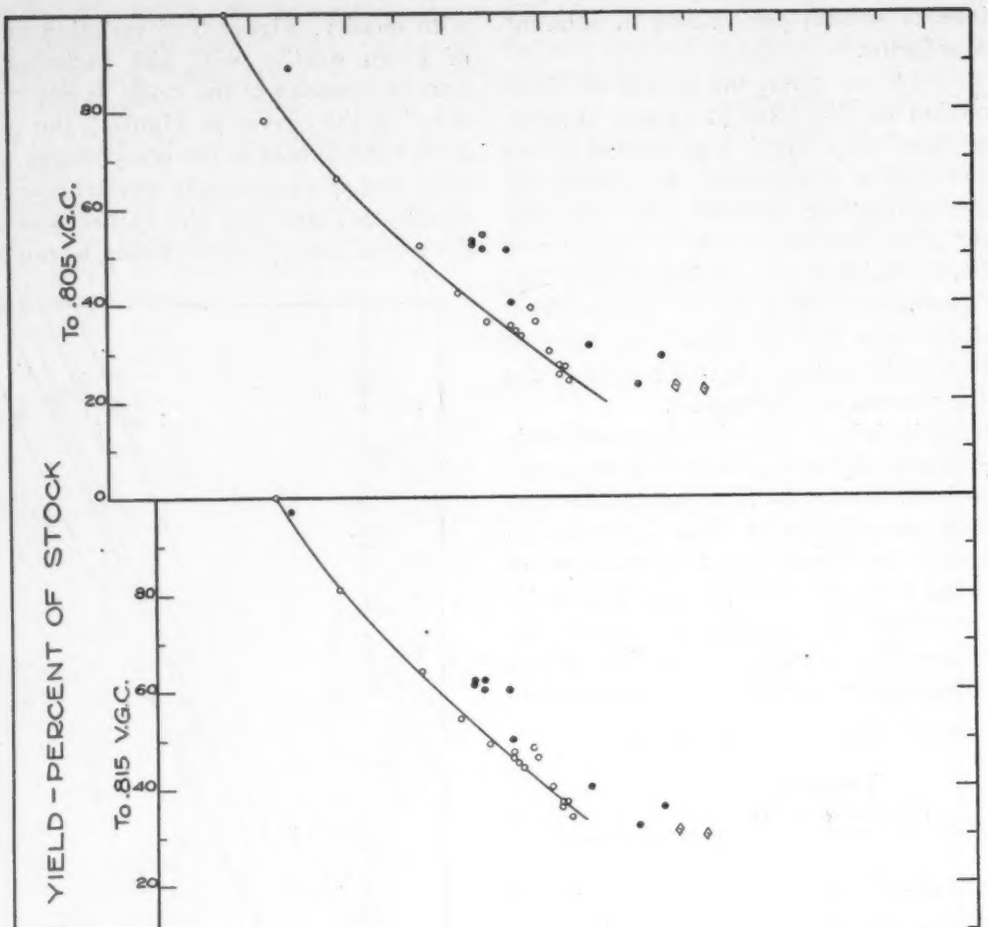

FIGURE 3

ative significance of each item. No items of burden are included.

Table 4 gives the consumption of steam, refrigeration, water, and power separately per unit volume of oil and of nitrobenzene charged; also the labor requirements and estimated repair costs per day for a 1000-barrel plant. Although extraction temperatures ranging from 41° to 77° F. have been used in the experiments cited, the refrigeration requirements have been uniformly estimated for a temperature of 50° F. In Table 5 the costs are summarized and combined to show the total cost of processing stocks requiring treatment with 1, 1.25, 1.50, 1.75, and 2.0 volumes of nitrobenzene per volume of charge.

COST OF SOLVENT LOSS

No better estimate of nitrobenzene consumption can be made now than was given in the paper on

TABLE 2
Stocks of Low Wax Content

Stock No.	Description	Treatment: Per Cent Nitrobenzene	Treatment: Temperature, °F.	Treatment: Nitraffin Yield*		Properties: Viscosity at 100° F.	Properties: Viscosity at 210° F.	Properties: Gravity, A.P.I.	Properties: Viscosity-Gravity Constant
2	Barber's Hill distillate	...	...	...	Stock	...	136	20.2	0.859
		130	50,59,68	54.0	Nitraffin	...	98	26.1	0.819
		130	59,68,77	46.0	Nitraffin	...	96	27.0	0.813
3	Barber's Hill distillate	...	...	...	Stock	...	73	21.1	0.864
		125	50	49.9	Nitraffin	...	59	27.9	0.818
9	Barber's Hill residuum	...	...	...	Stock	...	419	16.3	0.874
		150	59,77,95	40.0	Nitraffin	...	186	24.6	0.818
10	Coastal distillate	...	...	...	Stock	612	...	20.6	0.874
		100	50	27.9	Nitraffin	319	...	30.3	0.810
		150	50	21.6	Nitraffin	315	...	31.5	0.801
11	Solar stock	...	...	...	Stock	305	...	24.6	0.853
		136	50	44.0	Nitraffin	185	...	32.0	0.807
13	Dewaxed hydrogenated West-Texas distillate	...	...	...	Stock	431	57.5	27.1	0.828
		150	68	62.5	Nitraffin	...	56.6	30.8	8.803
14	Colombian distillate	...	...	...	Stock	946	70.0	20.9	0.868
		125	41	43.5	Nitraffin	484	...	28.7	0.814
15	Hydrogenated Colombian distillate	...	...	...	Stock	457	...	29.0	0.813
		150	77	77.5	Nitraffin	444	...	30.1	0.805
16	Mid-Continent distillate (dewaxed)	...	...	...	Stock	361	...	25.3	0.845
		150	41	51.8	Nitraffin	224	...	31.8	0.805
17	Winkler distillate	...	...	...	Stock	538	...	20.9	0.875
		150	41	29.5	Nitraffin	274	...	30.8	0.808
20	Winkler residuum	...	...	...	Stock	...	597	12.0	0.904
		150	41	28.0	Nitraffin	...	158	25.9	0.812
21	Barber's Hill residuum	...	...	...	Stock	...	454	16.0	0.876
		150	50,86,113	38.3	Nitraffin	...	196	24.4	0.819
23	Barber's Hill distillate	...	...	...	Stock	...	305	17.5	0.869
		120	68,77,86	42.0	Nitraffin	...	149	25.5	0.816
		150	68,77,86	41.5	Nitraffin	...	141	26.3	0.810
24	Barber's Hill distillate	...	...	...	Stock	...	420	16.5	0.872
		160	77,95,122	31.3	Nitraffin	...	151	26.3	0.809
25	Reduced pressed distillate	...	...	...	Stock	266	...	23.4	0.864
		150	41,45,50	42.6	Nitraffin	166	...	31.9	0.810
26	Reduced pressed distillate	...	...	...	Stock	476	...	22.2	0.866
		150	50	38.7	Nitraffin	249	...	31.0	0.809
27	Barber's Hill distillate	...	...	...	Stock	434	...	22.4	0.865
		150	41,59,77	43.7	Nitraffin	257	...	29.6	0.818
28	Winkler residuum	...	...	...	Stock	...	498	13.3	0.898
		175	41	32.2	Nitraffin	...	181	25.0	0.816

*Percent of stock by volume.

this process presented to the Institute by Ferris and Houghton last November*, viz., 0.08 percent of the volume of solvent applied to each volume of oil. The losses experienced in practice will depend upon engineering design and upon operating skill. Figures are given in Table 5 for each case, showing the cost of losses of from 0.1 percent to 0.2 percent. Since for an oil-solvent ratio of 1:1.5 this cost amounts to 30 percent of the total for the higher loss, a regrettably large margin of uncertainty is introduced which can only be eliminated after actual experience with this process over a considerable period, or possibly to some extent by the practical experience of other refiners with solvents of similar volatility, in similar processes. The variation in operating costs with variation in solvent-oil ratio is shown in Figure 5.

TOTAL DIRECT COSTS

Taking from Table 5 the average ratio of solvent to charge of 1.5 to 1, and 0.1 percent solvent loss, the relative importance of the various items of direct cost are:

	Cost Per 100-Gal. Charge	Percent of Total
Nitrobenzene loss	$0.13	17
Labor	0.23	31
Steam	0.18	25
Repairs	0.08	11
Refrigeration (including its own water, power, and steam)	0.07	10
Power	0.02	3
Water	0.02	3
Total	$0.73	100

Because of the great variation in nitraffin yields, both with change of stock and nitraffin quality, the cost per volume of stock has little meaning alone.

Figure 6 shows the variation in direct manufacturing cost of nitraffin with variation in the viscosity-gravity constant of stock for nitraffins of 0.800, 0.805,

*S. W. Ferris and W. F. Houghton, "The Nitrobenzene Process for Lubricating Oil," Proc. A. P. T. 13 (III), 82 (1932).

TABLE 3

Stocks of High Wax Content

Stock No.	Description	Treatment: Per Cent Nitrobenzene	Treatment: Temperature, °F.	Nitraffin Yield*		Properties: Viscosity at 100° F.	Properties: Viscosity at 210° F.	Properties: Gravity, A.P.I.	Viscosity-Gravity Constant
1	Oklahoma residuum	...	...	...	Stock	...	142	20.2	0.858
		100	41	58.5	Nitraffin	...	101	26.5	0.816
		150	41	55.0	Nitraffin	...	99	27.2	0.810
4	East-Texas distillate (partially dewaxed)	...	...	...	Stock	425	...	23.6	0.856
		125	50	50.0	Nitraffin	...	55	30.2	0.804
5	East-Texas distillate (partially dewaxed)	...	...	...	Stock	325	...	23.1	0.864
		100	50	41.4	Nitraffin	189	...	32.1	0.806
6	East-Texas distillate (partially dewaxed)	...	...	...	Stock	...	76.5	22.0	0.856
		125	50	53.0	Nitraffin	...	60	29.9	0.804
7	East-Texas distillate (partially dewaxed)	...	...	...	Stock	...	178	19.1	0.863
		150	50	52.0	Nitraffin	...	100	27.8	0.806
8	Hobbs distillate (partially dewaxed)	...	...	...	Stock	599	...	20.0	0.880
		125	41	30.5	Nitraffin	269	...	31.5	0.804
12	Pennsylvania residuum	...	...	...	Stock	...	220	24.4	0.818
		100	59	82.7	Nitraffin	...	188	27.4	0.797
18	Hobbs residuum	...	...	...	Stock	...	103	18.8	0.875
		125	41	15.5	Nitraffin	...	92	29.9	0.795
19	Hobbs residuum	...	...	...	Stock	...	275	14.6	0.895
		150	41	21.1	Nitraffin	...	105	28.7	0.798
22	Van Zandt distillate	...	...	...	Stock	1,266	69	22.1	0.858
		100	41	59.2	Nitraffin	...	56.6	29.2	0.811
25	Hobbs residuum	...	...	...	Stock	...	359	14.8	0.890
		150	41	24.8	Nitraffin	...	124	27.3	0.806

*Percent of stock by volume.

0.810, 0.815, 0.820, and 0.825 viscosity-gravity constant.

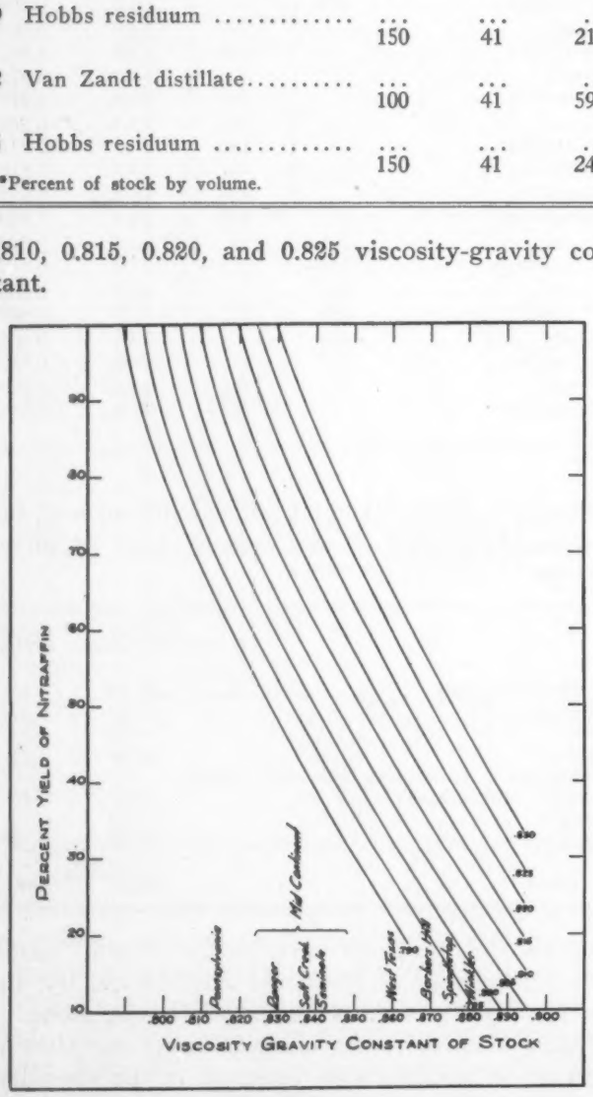

FIGURE 4

The cost data used in this figure are calculated throughout on the basis of the smoothed yield curves for low-wax stocks, and of a single solvent-oil ratio of 1.4 to 1. Since the actual ratios varied from 1:1 to 1.75:1, the latter basis may seem illogical; but it should be remembered that the actual ratios used are not of great significance, since in but few of the ex-

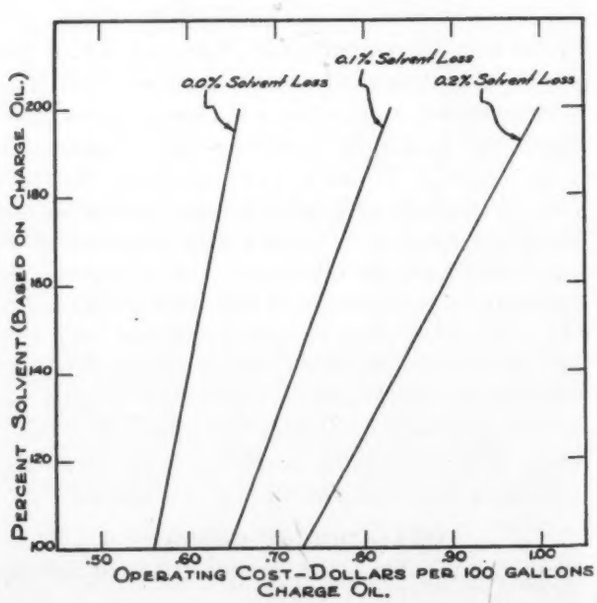
0.0% Solvent Loss
0.1% Solvent Loss
0.2% Solvent Loss
Percent Solvent (Based on Charge Oil.)
Operating Cost-Dollars per 100 Gallons Charge Oil.
.50 .60 .70 .80 .90 1.00

FIGURE 5

TABLE 4

Nitrobenezene Extraction—Service Requirements

	Dependent on Oil Throughput (Units Per 100 Gal. Oil Charge)		Dependent on Nitrobenzene Throughput (Units Per 100 Gal. Nitrobenzene Charge)	
Steam, lb.				
Heating	83		217	
Stripping	40			
Pumping	15		12	
Vacuum pumps	45		20	
General utility	50			
Total		233		249
Refrigeration, tons				
65 percent efficient	0.125	0.125	0.025	0.025
Water, gal.				
Cooling and conditioning	530		935	
Vacuum pumps	690		290	
Total		1,220		1,225
Power, kwh.				
Chillers and mixers	1.33			
Pumps	0.33		0.34	
Total		1.66		0.34

Independent of Throughput

Labor Position	Man Hours	Dollars Per Hour	Dollars Per Day
Foreman	8	1.25	10.00
Shift foreman	24	1.01	24.24
Stillman	24	0.89	21.40
Pumper	24	0.77	18.45
Refinery engineer	24	0.81	19.40
Laboratory assistant	24	0.76	18.20
Laborer	8	0.53	4.25
Total	136	Average 0.85	116.00

With 1000 bbl. per day oil charge—$0.232 per 100 gal. oil charge.

Repairs, 6.5 percent of $222,000 = $39.50 per day—0.079 per 100 gal. oil charge.

amples given was the optimum combination of extraction temperature and solvent ratio determined. In Table 6 the costs calculated from the actual data, and taken from the curves, are compared for low-wax stocks. The agreement, except in the case of the higher-asphaltic Winkler residuum, is generally within ± 10 percent, and sufficiently good to make the picture valid for the purpose intended. The actual cost of processing highly-asphaltic residuums of the type of Winkler is much lower than indicated by the curves.

The costs of nitraffin from the high-wax overhead stocks may be read from the curve with about the same deviation from the "actual" costs, by adding 0.010 to the desired viscosity-gravity constant of the nitraffin. This correction has a logical and significant background, since the nitraffins from these stocks have a viscosity-gravity constant about 0.010 higher after dewaxing than before. The increase in the constant in the case of low-wax stocks is comparatively insignificant, 0.002.

The quality of a finished oil most often required of a solvent process is 100 viscosity index. This is, in general, equivalent to a viscosity-gravity constant of 0.810. If viscosity-gravity constants of 0.840, 0.860, and 0.875 are taken as typical of Mid-Continent, West Texas, and Gulf Coast oils, then 100-viscosity-index-nitraffin yields and costs of the folowing order

TABLE 5

Nitrobenzene Extraction—Operating Costs

	Basis—100 Gal. Oil Charge			Basis—100 Gal. Nitrobenzene Charge		
Item	Quantity	Unit Cost	Cost	Quantity	Unit Cost	Cost
Steam	233 lb.	$0.30 per 1,000 lb.	0.0700	249 lb.	$0.30 per 1,000 lb.	0.0748
Refrigeration	0.125 tons	$0.45 per ton	0.0563	0.025 tons	$0.45 per ton	0.0113
Water	1,220 gal.	$6.00 per 1,000,000 gal.	0.0073	1,225 gal.	$6.00 per 1,000,000 gal.	0.0073
Power	1.66 kwh.	$0.01 per kwh.	0.0166	0.34 kwh.	$0.01 per kwh.	0.0034
Total			0.1502			0.0968
Labor			0.2320			
Repairs			0.0790			

Total Cost of Extraction per 100 Gal. of Oil Charge **Exclusive of Nitrobenzene Loss**

Volumes of nitrobenzene:	Dollars Per 100 Gal.				
Volume of oil	1.00	1.25	1.50	1.75	2.00
Item					
Steam	0.1448	0.1635	0.1822	0.2009	0.2196
Refrigeration	0.0676	0.0704	0.0733	0.0761	0.0789
Water	0.0146	0.0164	0.0183	0.0201	0.0219
Power	0.0200	0.0208	0.0217	0.0225	0.0234
Labor	0.2320	0.2320	0.2320	0.2320	0.2320
Repairs	0.0790	0.0790	0.0790	0.0790	0.0790
Total	0.5580	0.5821	0.6065	0.6306	0.6548
Nitrobenzene loss (at $0.085 per lb.):					
At 0.1 percent	0.0850	0.1063	0.1275	0.1486	0.1700
At 0.2 percent	0.1700	0.2125	0.2550	0.2975	0.3400
Grand Total:					
With 0.0 percent loss	0.5580	0.5821	0.6065	0.6306	0.6548
With 0.1 percent loss	0.6430	0.6884	0.7340	0.7792	0.8248
With 0.2 percent loss	0.7280	0.7946	0.8615	0.9281	0.9948

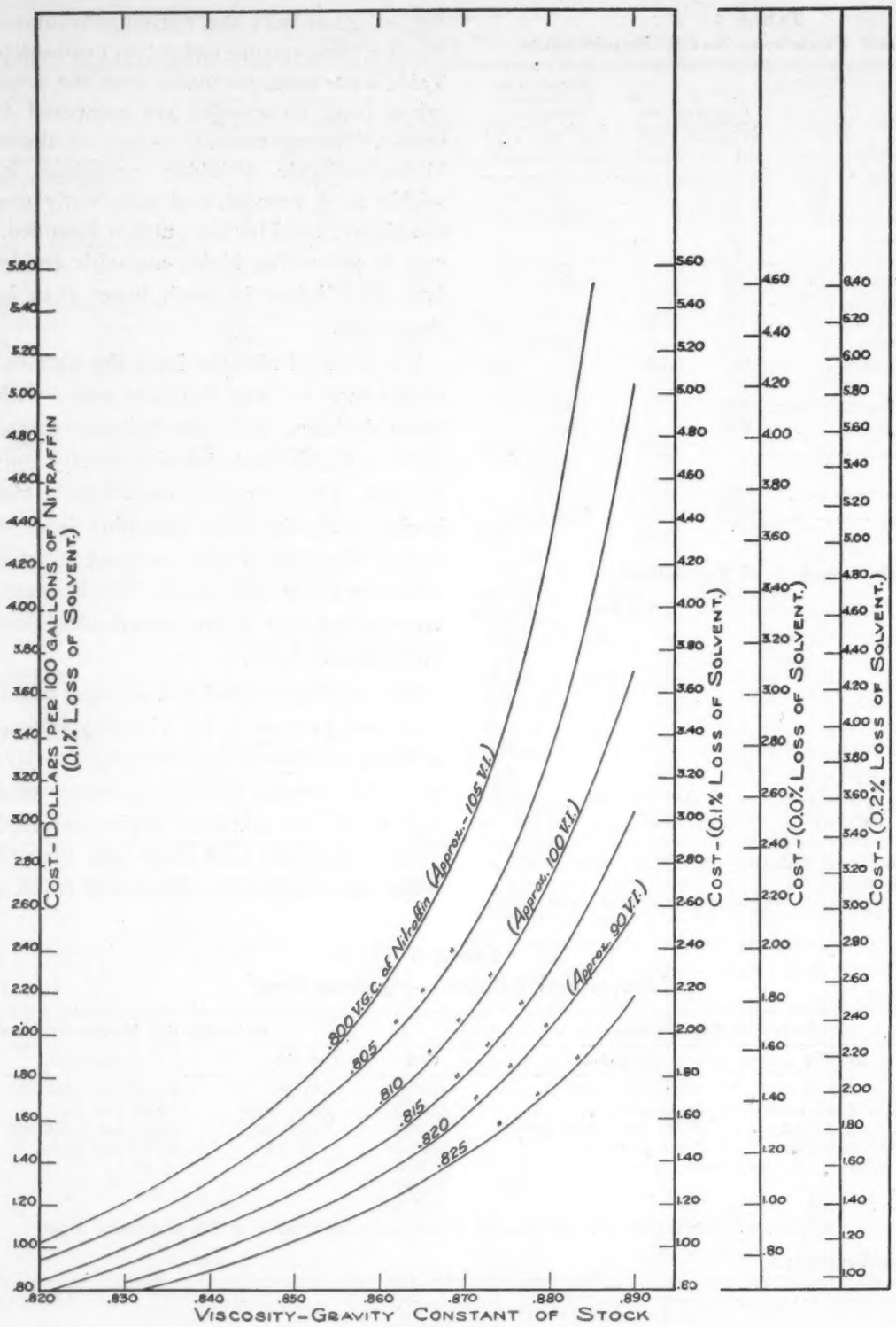
Cost - Dollars per 100 Gallons of Nitraffin (0.1% Loss of Solvent.)
Cost - (0.1% Loss of Solvent.)
Cost - (0.0% Loss of Solvent.)
Cost - (0.2% Loss of Solvent.)
.800 V.G.C. of Nitraffin (Approx. - 105 V.I.)
.805
.810 (Approx. 100 V.I.)
.815
.820
.825 (Approx. 90 V.I.)
Viscosity-Gravity Constant of Stock

FIGURE 6

from low-wax-content stocks may be expected:

	Mid-Continent	West Texas	Gulf Coast
Yield, percent	61	43	31
Manufacturing cost per 100 gal. of nitraffin.....	$1.17	$1.65	$2.28

DEWAXING

The effect of solvent extraction, irrespective of solvent used, is to concentrate in the solaffin the major part of the wax contained in the charge stock. Difficulty will be experienced in the dewaxing of those solaffins which are obtained in relatively low yield from a stock of normal or high wax content; otherwise the dewaxing costs applied to the finished oils will be normal. Obviously, the dewaxing process may be applied to stock or solaffin. The upper limit cost will be that occasioned by dewaxing the original stock to a sufficient extent to obtain the required pour point in the solaffin which, except in the case of

TABLE 6

Stocks of Low Wax Content (0.1 Percent Loss)

Stock No.	Viscosity-Gravity Constant of Stock	Description	Treatment Percent Nitrobenzene[1]	Viscosity-Gravity Constant of Nitraffin	Nitraffin Yield[3]		Manufacturing Cost Per 100 Gal. Nitraffin		Percent Deviation[2]
					Actual	From Fig. 4	From Actual Data	From 140 Percent Solvent	
2	0.859	Barbers Hill distillate	130	0.819	54.0	54.0	1.30	1.33	+2.3
			130	0.813	46.0	47.5	1.52	1.51	—0.7
3	0.864	Barbers Hill distillate.....	125	0.818	49.9	48.5	1.39	1.48	+6.5
9	0.874	Barbers Hill residuum.....	150	0.818	40.0	39.5	1.85	1.82	—1.6
10	0.874	Coastal distillate	100	0.810	27.9	31.6	2.31	2.27	—1.7
			150	0.801	21.6	22.5	3.42	3.19	—6.7
11	0.853	Solar stock	136	0.807	44.0	45.0	1.61	1.59	—1.2
13	0.828	Dewaxed hydrogenated West-Texas distillate	150	0.803	62.5	64.6	1.18	1.11	—6.0
14	0.868	Colombian distillate	125	0.814	43.5	40.5	1.59	1.77	+11.3
15	0.813	Hydrogenated Colombian distillate	150	0.805	77.5	84.6	0.95	0.85	—10.5
16	0.845	Dewaxed Mid-Continent distillate	150	0.805	51.8	50.8	1.42	1.41	—0.7
17	0.875	Winkler distillate	150	0.808	29.5	29.0	2.50	2.48	—0.8
20	0.904	Winkler residuum	150	0.812	28.0	12.0	2.64	5.98	+127
21	0.876	Barbers Hill residuum	150	0.819	38.3	38.6	1.93	1.86	—3.6
23	0.869	Barbers Hill distillate	120	0.816	42.0	41.5	1.63	1.73	+6.1
			150	0.810	41.5	35.5	1.78	2.02	+13.5
24	0.872	Barbers Hill distillate	160	0.809	31.3	32.2	2.42	2.23	—7.8
25	0.864	Reduced pressed distillate....	150	0.810	42.6	40.0	1.73	1.79	+3.5
26	0.866	Reduced pressed distillate....	150	0.809	38.7	37.1	1.90	1.94	+2.1
27	0.865	Barbers Hill distillate	150	0.818	43.7	47.5	1.69	1.51	—10.7
28	0.898	Winkler residuum	175	0.816	32.2	18.5	2.44	4.12	+69

[1] For calculated cost, 140 percent solvent used in each instance.
[2] $\frac{\text{(Cost from curves)—(cost from actual data)}}{\text{(cost from actual data)}} \times 100.$
[3] Percent of stock by volume.

TABLE 7

Stocks of High Wax Content (0.1 Percent Loss)

Stock No.	Viscosity-Gravity Constant of Stock	Description	Treatment Percent Nitrobenzene[1]	Viscosity-Gravity Constant of Nitraffin	Nitraffin Yield[3]		Manufacturing Cost Per 100 Gal. Nitraffin		Percent Deviation[2]
					Actual	From Fig. 4	From Actual Data	From 140 Percent Solvent	
1	0.858	Oklahoma residuum	100	0.816	58.5	62.0	1.10	1.16	+5.5
			150	0.810	55.0	56.3	1.34	1.28	—4.5
4	0.856	East Texas distillate (partially dewaxed)	125	0.804	50.0	51.3	1.38	1.40	+1.5
5	0.864	East Texas distillate (partially dewaxed)	100	0.806	41.4	46.0	1.55	1.56	+0.7
6	0.856	East Texas distillate (partially dewaxed)	125	0.804	53.0	51.0	1.31	1.41	+7.6
7	0.863	East Texas distillate (partially dewaxed)	150	0.806	52.0	47.0	1.42	1.53	+7.8
8	0.880	Hobbs distillate (partially dewaxed)	125	0.804	30.5	30.5	2.27	2.36	+4.0
12	0.818	Pennsylvania residuum	100	0.797	82.7	80.0	0.78	0.90	+15.4
18	0.890	Hobbs residuum	150	0.806	24.8	24.6	2.98	2.92	—2.0
19	0.895	Hobbs residuum	150	0.798	21.1	13.5	3.50	5.31	+51.8
22	0.858	Van Zandt distillate	100	0.811	59.2	57.1	1.09	1.26	+15.6

[1] For calculated cost, 140 percent solvent used in each instance.
[2] $\frac{\text{(Cost from curves)—cost from actual data)}}{\text{(cost from actual data)}} \times 100.$
[3] Percent of stock by volume.

TABLE 8

Acid Treated Stocks or Pennsylvania Stocks

Stocks	Viscosity	Color		Tons of Clay Per Barrel of Finished Oil
		Charge	Finished Oil	
Pennsylvania 153 oil.....	150 at 100°F.	4¾-5 (102-120 OD)	2¾-3 (18.6-24.4 OD)	0.01986
Pennsylvania 183 oil.....	180 at 100°F.	5¼-5½ NPA (140-162 OD)	2¾-3 (18.6-24.4 OD)	0.02673
Pennsylvania-FFF Valve	140-150 at 210°F.	5DD NPA (5340 OD)	7-7½ NPA (344-425 OD)	0.4324
Hivis	105-115 at 210°F.	8 NPA (520 OD)	7-7½ NPA (344-425 OD)	0.04545

Nitraffins

Stocks	Viscosity	Color		Tons of Clay Per Barrel of Finished Oil
		Charge	Finished Oil	
Residuum from pressed distillate..	162 at 100°F.	2¾ NPA (18.6 OD)	1½-1¾ NPA (2.8-4.5 OD)	0.008854
Residuum from pressed distillate..	231 at 100°F.	3¼ NPA (31.2 OD)	1¾-2 NPA (4.5-6.8 OD)	0.01043
Barbers Hill residuum.....	159 at 210°F.	3¼ + DDD NPA (9640 OD)	7 NPA (344 OD)	0.4073
Barbers Hill distillate.....	137 at 210°F.	4¾ + D NPA (744 OD)	7 NPA (344 OD)	0.07555

All yields based on No. 1 Clay.

complete removal, will always be higher than that of the stock. It will probably always be found less expensive to dewax the solaffin than the charge, if that can be done in one operation. There can be little question that the wide adoption of solvent-extraction processes, coupled with the trend towards narrow-boiling distillate motor oils, will add marked emphasis to the need for a feasible dewaxing procedure relatively insensitive to wax concentration and crystal form, and capable of sharply separating wax and oil.

DECOLORIZING

Nitrobenzene removes a sufficiently high proportion of the colored bodies from the nitraffins of both distillate and residual stocks to render acid treatment unnecessary. The cost of solvent treatment is thus appreciably offset—particularly in the case of residual stocks and in view of the real value of the nitrenes and the negligible value of acid sludges. Before treating with clay the nitraffins from residuums, it is necessary to allow them to stand after dilution and to remove about one percent of coloring matter which settles as a lower layer. Table 8 compares the clay consumption for two typical distillates and one residuum from Pennsylvania crude, with nitraffins of corresponding viscosities—the heavy nitraffin being made from a Barbers Hill residuum. In all three cases the clay consumption is lower for the nitraffin. A comparison is also made of the clay consumption of a mixed-base distillate, 105 sec. to 115 sec. viscosity, Saybolt Universal, at 210° F., treated almost to desired color with acid; and a nitraffin from an overhead distillate, 137 sec. viscosity, Saybolt Universal at 210° F., which had received no acid treatment. While the clay consumption for the nitraffin is higher, it will be noted that it represents a yield of 225 barrels per 17-ton filter. In all cases no differences could be observed in the color stability of the oils.

The Chlorex Process *for Producing High-Grade* Lubricating Oil

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CHLOREX (β, β'-dichloroethyl ether) has been shown to be a very selective solvent for production of high-grade lubricating oils. In addition to its selectivity the other properties of this solvent are such that it is very well suited to commercial application. Its boiling point (353° F.) is well below the initial vaporization temperature of lubricating oils, and this permits ready recovery of the solvent by distillation. Its vapor pressure at atmospheric temperatures is so low that need for protection against evaporization loss is minimized. Its freezing point is so low that precautions in this regard are unnecessary. Due to its high density and low viscosity, separation of raffinate and extract solutions is rapid.

The Chlorex process has been applied to commercial production of lubricating oils. Three plants are operating with the process. These plants have a combined capacity of 1400 barrels per day. This first plant built has been in operation more than a year.

In operation, Chlorex and oil are passed countercurrently in a series of mixing and settling stages, and the final raffinate and extract solutions from these stages are sent to stills for separation of oil and solvent. Distillation may be carried out at atmospheric pressure and in the presence of steam. Chlorex is only slightly soluble in water, but for economic operation the solvent dissolved in the condensed steam used for distillation is recovered. Convenient and efficient methods of recovery have been developed.

This paper gives data on operating requirements, and from these the costs may be estimated for given unit costs. In general, cost of operation should prove to be low.

Experience with operating the Chlorex process has shown that it is well adapted to producing the high grade of lubricating oil required for the modern automobile.

This paper was presented at Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933.

I. INTRODUCTION

THE present symposium attests to the interest of refiners in developing lubricating oils which fulfill the exacting requirements of the modern automobile. A background of research has pointed out the characteristics necessary for oils to fulfill these requirements, and this research has resulted in emphasizing the following desirable characteristics of motor oils:

1. The rate of change of viscosity with temperature should be small.
2. The A. S. T. M. pour point should be below that temperature at which engine starting will be expected.
3. The oil should deposit a minimum of engine carbon in use.
4. The resistance to sludging should be sufficient to protect the engine against hazards of sludge formation.

Oils excelling in all four of these characteristics provide the motorist with assurance of satisfactory lubrication under all conditions of operation. Moreover, recent researches[1-2] have shown that the inclusion of the characteristics of small change in viscosity with temperature and high degree of resistance to sludging provide the very advantageous characteristic of low-oil consumption.

It was for the purpose of manufacturing such a superior line of oils that this company developed and applied two new processes, one of which is the Chlorex process of solvent extraction. Through its use, oils of high viscosity index† and resistance to sludging are obtained. Operated on distillate oils, it assures a minimum of engine carbon formation[3]; and, in conjunction with propane dewaxing[4], oils excelling in all four important points mentioned above are produced.

The general advantages of Chlorex (β, β'-dichloroethyl ether) as a selective solvent for the production of super-quality motor oils from various representative crudes was dealt with in a recent paper[5] presented by J. M. Page, Jr., C. C. Buchler, and S. H. Diggs at the spring meeting of the American Chemical Society. The principal purpose of the present paper is to describe the application of this solvent to the commercial production of lubricating oils of high quality.

II. SELECTIVITY OF CHLOREX AND ITS APPLICABILITY TO VARIOUS STOCKS

A brief summary of the information previously published will serve to show the general character-

† Viscosity-index system of Dean and Davis, *Chem. Met End.*, 36, 618 (1929); see also Davis, Lapeyrouse, and Dean, *Oil Gas J.*, 30, no. 4, 92 (1932), is used throughout this paper. For simplicity, viscosity index is abbreviated to v.-i in the following sections.

istics of Chlorex which are relative to its suitability as a solvent for production of high-grade motor oil. Such a review is given in the following few paragraphs.

From the viewpoint of selectivity, the ideal solvent will separate completely the undesirable, low v.-i., fraction from the desirable, high v.-i., fraction. To do this it must possess high-solvent power for the undesirable fraction and dissolve none of the desired fraction at the temperature of extraction.

The vast majority of available solvents fails to attain to these ideals in one of two respects: either, though miscible in all proportions with the undesirable components, they possess too high a solubility for the desirable components; or, though satisfactorily low in solvent power for the desirable components, they at the same time fail in possessing complete miscibility in all proportions with the undesirables. Solvents of the first type will give good v.-i. raffinates, it is true, but at the expense of loss of good components due to their excessive solubility in the extract phase; solvents of the second type, in general, do not economically attain to the best v.-i. raffinates because of an insufficent degree of difference in partition ratios of the desirable and undesirable fractions between raffinate and extract phases.

The excellent suitability of Chlorex as a selective solvent, when considered according to the above viewpoint, is illustrated in Table 1, in which the solubility of 100 v.-i. (S.A.E. 20) oil and 0 v.-i. (S.A.E. 20) oil in Chlorex is shown For comparison the solubilities of the same oils in nitrobenzene and in acetone are also shown

From these data it may be readily seen that Chlorex possesses solvent properties for high and low v.-i. oils which combine the better properties of each of the two major classifications, into which most other solvents in general may be grouped.

Investigation has indicated that Chlorex is suitable to the extraction of all types of stocks. Table 2 gives results of Chlorex extraction of oils from various sources, particularly with reference to possible improvement in quality. Consideration of these results leads to the conclusion that the solvent will produce a substantial improvement over the quality of the oils generally marketed from any given crude source. With reference to Table 2, special mention should perhaps be made to the improvement in resistance to sludging. The data in Table 2 bearing on this point were obtained by the Indiana oxidation test[1]. In this test the oil is retained at 341° F., while air is bubbled through it at a measured rate. Samples of the oil are then removed at frequent intervals, and the percent asphaltenes formed by the oxidation are determined on these samples. From these data the time required to form 0.1 percent asphaltenes is determined, and

TABLE 1

Solubilities of 100 and of 0 V.-I. Oil in Various Solvents

Temperature (°F.)	(Solubility in cc. per 100 cc. solvent) 100 V.-I. Stock			0 V.-I. Stock		
	Nitrobenzene	Acetone	Chlorex	Nitrobenzene	Acetone	Chlorex
40	13	1.2	1.1	Miscible in all proportions	5.0	Miscible in all proportions
60	19	2.4	2.2	Miscible in all proportions	6.8	Miscible in all proportions
80	25	3.7	3.8	Miscible in all proportions	11.0	Miscible in all proportions

TABLE 2

Improvement in Quality Obtained by Chlorex Extraction of Oils from Various Sources

Sample	Source	Viscosity at 210° F.	Total Chlorex Used (Per Cent by Volume of Oil Charged)	Before Extraction			After Extraction		
				Viscosity Index	Sludging Time* (Hours)	Carbon Residue (Conradson)	Viscosity Index	Sludging Time* (Hours)	Carbon Residue (Conradson)
1	Mid Continent	50	250	75	21	0.05	96	71	0.02
2	Mid Continent	50	300	75	20	0.05	102	110	0.03
3	Mid Continent	90	250	75	37	0.78	85	107	0.21
4	Mid Continent	90	250	75	37	0.78	90	127	0.21
5	Mid Continent	183	250	83	64	2.69	92	240	1.19
6	Pennsylvania	50.5	200	103	30	0.17	117	180	0.13
7	California	59	600	54	10	0.64	95	83	0.27
8	West Texas	64	250	64	29	0.03	108	74	0.002

* Indiana oxidation test—70 hours and longer represent such quality as to lead to no serious sludge formation when the oil is used under severe conditions in an engine.

this is called the "sludging time." The sludging time as determined by this test has been correlated with performance of the oil in an engine, and it has been found that use of an oil having a sludging time of 70 hours or more will lead to no serious sludge formation in the engine, even under severe conditions.

Although plant application of the Chlorex-extraction process for commercial reasons has up to now been limited to distillate stocks, extensive laboratory investigation on residual stocks, covering the range of viscosity from heavy motor oils through bright stocks, has shown beyond question the adaptability of this process to stocks of as widely different properties from Pennsylvania, Mid-Continent, and West Texas residuums.

Although Pennsylvania residual stocks have initially a good temperature coefficient of viscosity (high viscosity index), Chlorex extraction noticeably improves even this property. The value of extraction, however, in the case of Pennsylvania stocks lies not in this direction primarily, but rather in the fact that such extraction removes a small quantity of highly undesirable constituents whose deleterious effect on engine performance is shown by the pronounced improvement in the sludging resistance of the finished oil.

Oils having viscosity indices equal to Pennsylvania oil, and extremely stable toward sludging, have been prepared with very good yields from Mid-Continent and Texas residuums by Chlorex extraction, followed by percolation through clay. Thus, counter-current use of about two volumes of Chlorex on these residuums produces oils of 95 to 100 viscosity index which are extremely stable.

The extraction process on residuums may sometimes be combined to advantage with liquid-propane treatment to remove asphalt before or during extraction. This treatment with propane gives better color and/or increased percolation yield on treating with clay to desired color.

III. SUITABILITY OF CHLOREX TO COMMERCIAL APPLICATION

The application of Chlorex to commercial production can be carried out with a minimum of investment. Optimum extraction temperatures are in general not greatly different from the normal atmospheric temperature, so that investment and operating cost for control of extraction temperature is extremely low. Its other properties, also, as shown in Table 3, make it particularly suited to plant use.

TABLE 3
Properties of Dichloroethyl Ether

Structural formula	$Cl\text{-}CH_2\text{-}CH_2\text{-}O\text{-}CH_2\text{-}CH_2\text{-}Cl$
Boiling point, °F.	352.4
Freezing point, °F.	—61
Specific gravity, 20°/20°C.	1.222
Viscosity at 78°F., centipoises	2.06
Viscosity at 77°F., Saybolt Universal	32
Viscosity at 32°F., Saybolt Universal	39
Vapor pressure at 100°F., mm.	10
Latent heat of vaporization, B.t.u. per lb.	115.4
Specific heat at 85°F.	0.369
Flash point (closed cup), °F.	168
Solubility in water at 20°C., percent	1.01
Solubility in water at 90°C., percent	1.71

The high specific gravity of the solvent makes separation rapid, so that small-sized separating equipment is required. Its low viscosity is also a factor of importance in aiding separation. A small amount dissolved in a viscous-base oil also cuts the viscosity of the latter quite sharply. Its boiling point is well below the initial vaporization temperature of lubricating oils, so that recovery is readily accomplished by distillation. Its low vapor pressure at extraction and storage temperatures contributes to low construction costs and minimizes solvent losses due to evaporation. Its freezing point is so low that precautions in this regard are unnecessary. It is noninflammable at ordinary temperatures; the flash point is well above that normally considered hazardous in petroleum refineries. Its solubility in water is sufficiently low to permit distillation with open steam. When the amounts of steam used are large enough to justify it, the dissolved Chlorex substantially can be completely recovered from the water by one of several alternative methods described hereafter.

Chlorex has now been in plant use for over a year and in laboratory use for a much longer period, and in no case has there been any evidence of sickness or injury due to its use.

IV. GENERAL EXTRACTION CONDITIONS

The effects of temperature and solvent ratio upon the yield and viscosity index of the raffinate is illustrated in Figure 1 which shows the results of batch extraction of a Mid-Continent distillate (S.A.E. 50). The solid lines represent relationships existing between yield and viscosity index at constant solvent

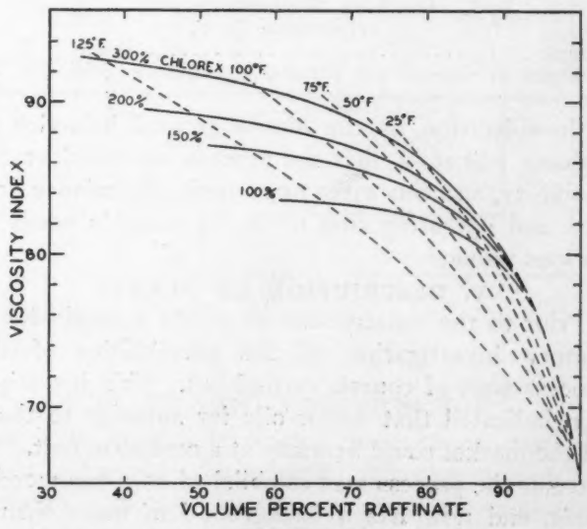

FIGURE 1
Effect of quantity of solvent and temperature of extraction on yield of oil of given viscosity index. Batch Extraction.

ratio, and the broken lines connect points of constant temperature. In general, the yield of oil of a given viscosity index, and the solvent ratio required to produce it, increases with decreasing temperature.

It follows from the foregoing that the solvent required per volume of finished oil of a given viscosity index on batch extraction is also a function of temperature; this is illustrated in Table 4 showing the relationship for production of an oil of 88 viscosity index (computed from data in Figure 1).

TABLE 4
Volumes of Solvent Required Per Volume of Finished Oil

Batch Extraction to 88 V.I. Computed from Data in Figure 1.				
Temperature, °F.	125	100	75	50
Volumes of solvent per volume of raffinate	3.34	3.3	3.64	4.13

The results given in Figure 1 and Table 4 are merely for illustrative purpose to show the general effect of temperature and solvent ratio when using the same method of extraction, which in this case is single-batch extraction. The solvent required under these extraction conditions is a maximum. Through the use of a counter-current extraction method the solvent required is substantially less than that shown above. Moreover, the amount of solvent required to produce a given result as to viscosity index in a counter-current system decreases as the number of stages increases. This is illustrated in Table 5, where the effect of the use of 2 and 4 stages is shown in comparison with that for batch when making an 88 v.-i. raffinate from a Mid-Continent distillate (S.A.E. 50).

TABLE 5
Volumes of Solvent Required Per Volume of Finished Oil—88 V. I.

Batch vs. Counter-current Temperature, 75°F.			
Stages	Batch	2	4
Volumes of solvent per volume of raffinate	3.64	2.8	1.4

Consideration of the above general behavior of Chlorex will show that the process has considerable flexibility, and this gives opportunity to balance first cost and operating cost to fit the possible needs of various refiners.

V. DESCRIPTION OF PLANTS

Prior to the construction of plants a detailed laboratory investigation of the possibilities of the process was, of course, carried out. This investigation indicated that motor oils far superior to those on the market could be made at a moderate cost. As a result the process was established on a commercial basis, and now has a background of more than a year's commercial operation. Experience has shown that solvent ratios, required yields, and quality of finished oil have all checked very closely with those predicted from laboratory data. Solvent losses have been lower than was assumed when constructing the first plant, averaging about 0.2 percent per use.

Three plants have been put in commercial operation, charging capacities of these plants being 800, 500, and 100 barrels per day. They are located at Wood River, Illinois; Casper, Wyoming, and Tulsa, Oklahoma. While they all follow the same general principles, they differ somewhat as to details of the various operations; thus in one plant distillation for solvent recovery is carried out in a pipe still, in a second plant the distillation is carried out in shell stills, while the third employs vacuum supplemented by flashing to atmospheric and stripping with steam. The method of extraction also differs; thus one plant employs a compact continuous counter-current extractor which is being developed to simplify this phase of the operation; a second started operation on a batch-extraction basis, but now employs three continuous counter-current stages of the type customarily proposed for solvent-extraction processes; and a third employs single-batch extraction, although plans are being completed for installation of continuous counter-current stages. Two of the plants handle wax-bearing stock, while the other handles a dewaxed charge. These detailed differences in the three plants were made for the purpose of getting operating information to permit prompt determination of the most satisfactory type of plant for future construction.

A general flow diagram of the process is shown in Figure 2. Oil is charged from the storage tank **A**, and is extracted counter-currently with Chlorex from storage tank **B**; for the purpose of this illustration, three-stage counter-current equipment is shown. The charge oil is mixed with twice-used extract from the separating drum **E-2**, and the mixture passed through the exchanger **C** to regulate the temperature for extraction. The mixture is then passed through the mixer **D-1** and into separating drum **E-1**. The extract solution from **E-1** passes to the intermediate solution storage tank **F**. Raffinate solution from **E-1** is mixed in **D-2** with extract from **E-3** and passed to the separator drum **E-2**. The raffinate solution from this stage is then mixed in **D-3** with fresh Chlorex from **B** and passed into **E-3**. The raffinate solution from **E-3** is then stored in the intermediate storage tank **G**. The extract and raffinate solutions are charged to their respective stills **H** and **J**. The solvent-free oils from these stills are passed to the final extract and raffinate storage tanks **O** and **P**. Chlorex and steam are condensed in the condensers **K-1** and **K-2**, and separated from one another in **L-1** and **L-2**—the Chlorex returning to the storage tank **B** while the water is collected in the intermediate storage tank **M**, from where it is heated in the still **N** and about 10 percent of it evaporated—the dissolved Chlorex being recovered as distillate. The denuded water is discarded to the sewer while the distillate is condensed in **K-3** and separated in **L-3**, the small amount

of distillate water being re-cycled while the Chlorex is returned to **B.**

With regard to Figure 2 it should be noted that heat exchange can be incorporated in the plant at appropriate points, especially when the method of distillation is continuous.

Common materials of construction are used throughout, and fabrication to stand only moderate pumping pressures is sufficient. Flow controllers on the oil and Chlorex streams to the extraction equipment have been found satisfactory and convenient. Float controls on the separator drums are used, and this method has also been found satisfactory. The mixing of oil and Chlorex in the extraction is done by an open impeller-type mixing pump.

Figure 3 is a photograph of the Chlorex plant at Casper, Wyoming, showing Chlorex storage tanks (right rear), building for housing pumping equipment, and (left foreground) solution and finished oil storage.

Figure 4 is a photograph of the Wood River plant. Chlorex storage and the oil cooler are shown in the right and center foreground, respectively. Behind the cooler is shown the counter-current contacting equipment and the pump house. Stills for the recovery of the solvent are part of the battery in the background.

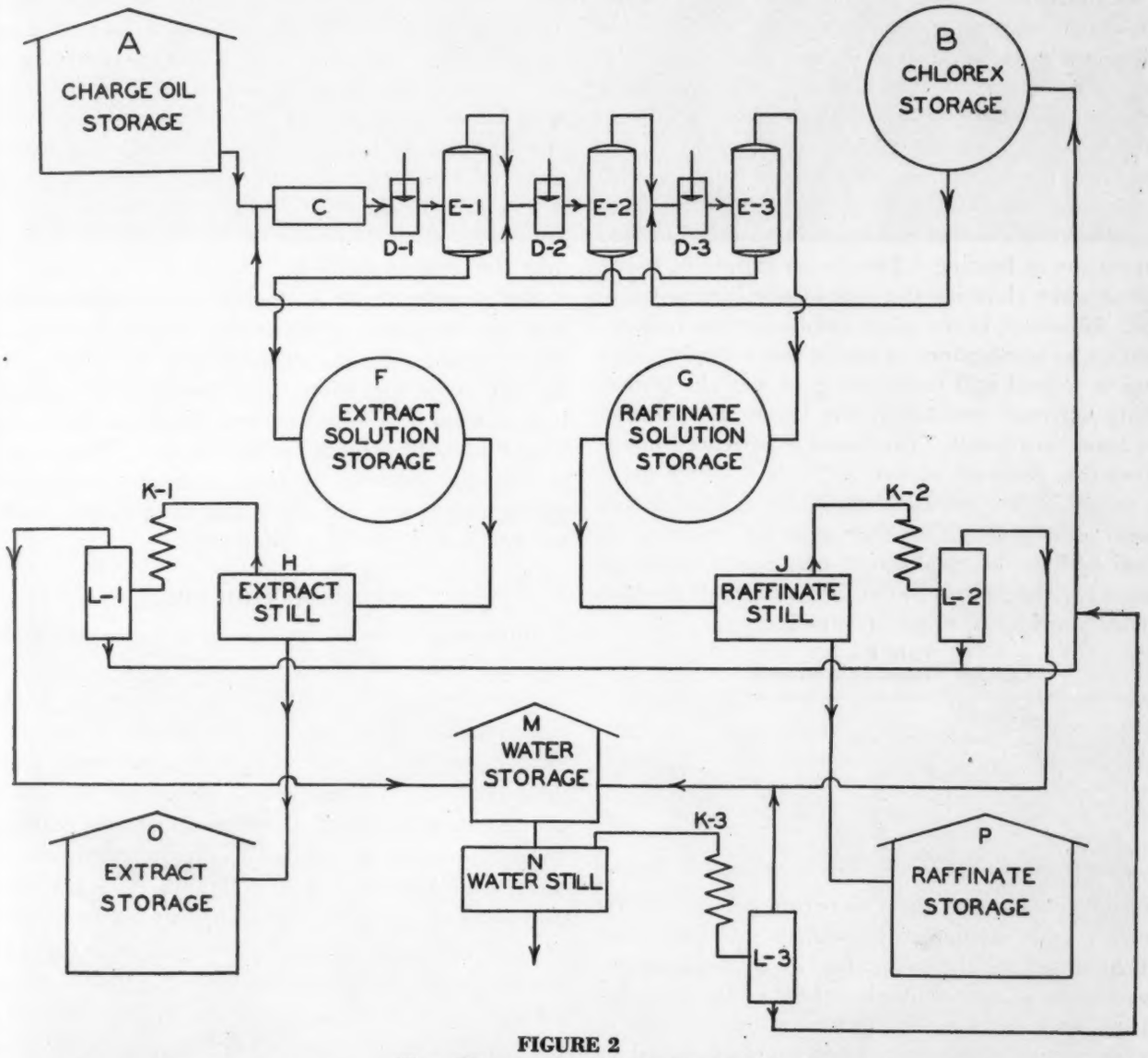

FIGURE 2
Flow Diagram of Chlorex Process

VI. SOLVENT CONSERVATION

In any extraction process precautions must be taken to conserve the solvent. A background of experience with the Chlorex process has brought out certain features which are worth noting.

Relatively large plants can operate with the Chlorex process on an astonishingly small solvent storage. The high density of the solvent contributes to rapid separation, which minimizes the amount in process. Continuous methods of distillation, where

solutions are heated in tubes, further contribute to low retention. Thus the solvent required to operate a plant processing about 40,000 gallons of oil a day is about 10,000 gallons of solvent. More solvent is, however, frequently a convenience.

The low vapor pressure of Chlorex at the temperatures of extraction minimizes the necessity of protection against evaporation. Plant experience in handling this solvent has verified the fact that Chlorex is unusually stable in comparison with other chlorinated compounds. Under normal temperatures no detectable decomposition occurs. Under the elevated temperatures of distillation a minute amount of decomposition occurs; the small amount of acid so formed is neutralized by the introduction of ammonia into the vapor line entering the condenser.

Laboratory investigation has shown that the decomposition of Chlorex is a function of the time and temperature of heating. This factor should be borne in mind when choosing the type of distillation equipment. However, in the plant employing batch distillation under atmospheric pressure and stripping with steam to a final still temperature of 400° F. (representing extreme conditions) the losses in operation have been very small. The losses experienced in operating this plant are shown in Table 6, which gives the record of five months which are typical of the present development of that type of process. It should perhaps be emphasized that losses shown in Table 6 are the summation of losses from all sources, as determined by monthly inventories.

TABLE 6
Chlorex Losses—All Sources

Month	Chlorex Processed (Gallons)	Chlorex Loss as Percent of Chlorex Processed (Percent)
1	1,370,630	0.2571
2	1,703,950	0.1103
3	1,870,000	0.2017
4	1,332,130	0.1833
5	2,268,940	0.1251

As indicated above, there is reason to believe that pipe-still and vacuum-still methods of distillation lead to a substantial reduction in over-all losses; however, the plants employing these methods of distillation have not been in operation long enough to give as certain a statistical monthly loss record as that of Table 6.

As stated above, Chlorex dissolved in the water is recovered by distillation of a relatively small amount of the water. Flash distillation is desirable, and at least 90 percent of the dissolved Chlorex can be recovered in the condensate on evaporation of 10 percent of the water. Two alternate methods of recovery of Chlorex from water have been proposed and investigated in the laboratory. These are: 1, injection of the water into the steam line leading to the still and subsequent removal of the so heated water before entering the still; thus the water is heated and denuded, then removed—the Chlorex returning to the still with the steam; and, 2, contacting the water with the incoming charge of oil. Both these methods, after laboratory investigation, appear as efficient as the present developed method of recovery by distillation of a portion of the water, and there is reason to believe that they can be developed to improve over the present method.

The oil products from the process are substantially free of chlorides. Quantitative determinations of total chlorides in the reduced oils have been frequently made, and these have shown a chloride content of from 0 to 0.0017 percent for the raffinate and from 0 to 0.010 percent for the extract. These traces are wholly removed by the subsequent percolation through clay, and a positive test of chlorides cannot be obtained on the percolated oils.

VII. OPERATING COSTS

Operating costs are perhaps best indicated by tabulation of the requirements of the various items affecting direct costs. For this purpose let us assume operation of a plant such as that shown in Figure 2, charging 50,000 gallons of oil daily and using a solvent ratio of 1.5 volumes of Chlorex per volume of oil. Since a choice of distillation methods exists, it seems desirable to set forth the requirements for alternative methods of distillation. The estimated requirements for such a plant are shown in Table 7.

Fixed charges for plant investment have been omitted from the above tabulation for the following reasons: The process is so readily adapted to use of the usual refinery equipment that in many cases plants of large capacity can be obtained at a rela-

TABLE 7
Estimated Daily Requirements—Direct
(Charging 50,000 gallons oil daily and 75,000 gallons Chlorex daily)

Item	Requirements: Plant Using Pipe Still for Distillation	Requirements: Plant Using Batch Stills for Distillation
Labor	2 to 3 men pershift	2 to 3 men per shift
Steam, lb.	50,000	275,000
Fuel oil, gal.	2,200	2,600
Water, gal.	700,000	1,000,000
Power, kwh.	720	250
Solvent loss, gal.	Not more than 150, probably less (see Table 4)	Not more than 150, probably less (see Table 4)

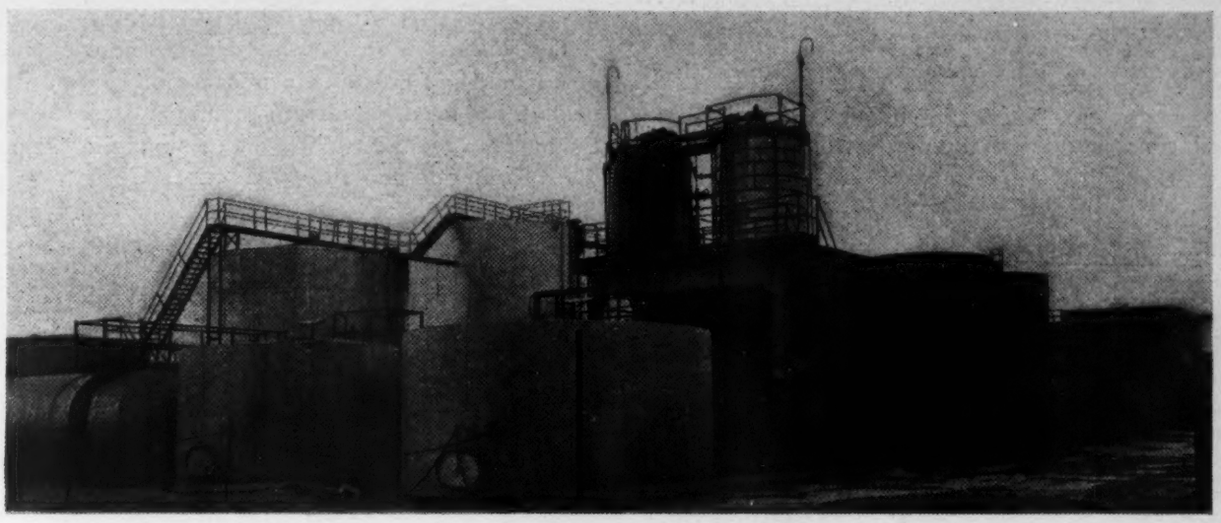

FIGURE 3
Chlorex Plant at Casper, Wyoming

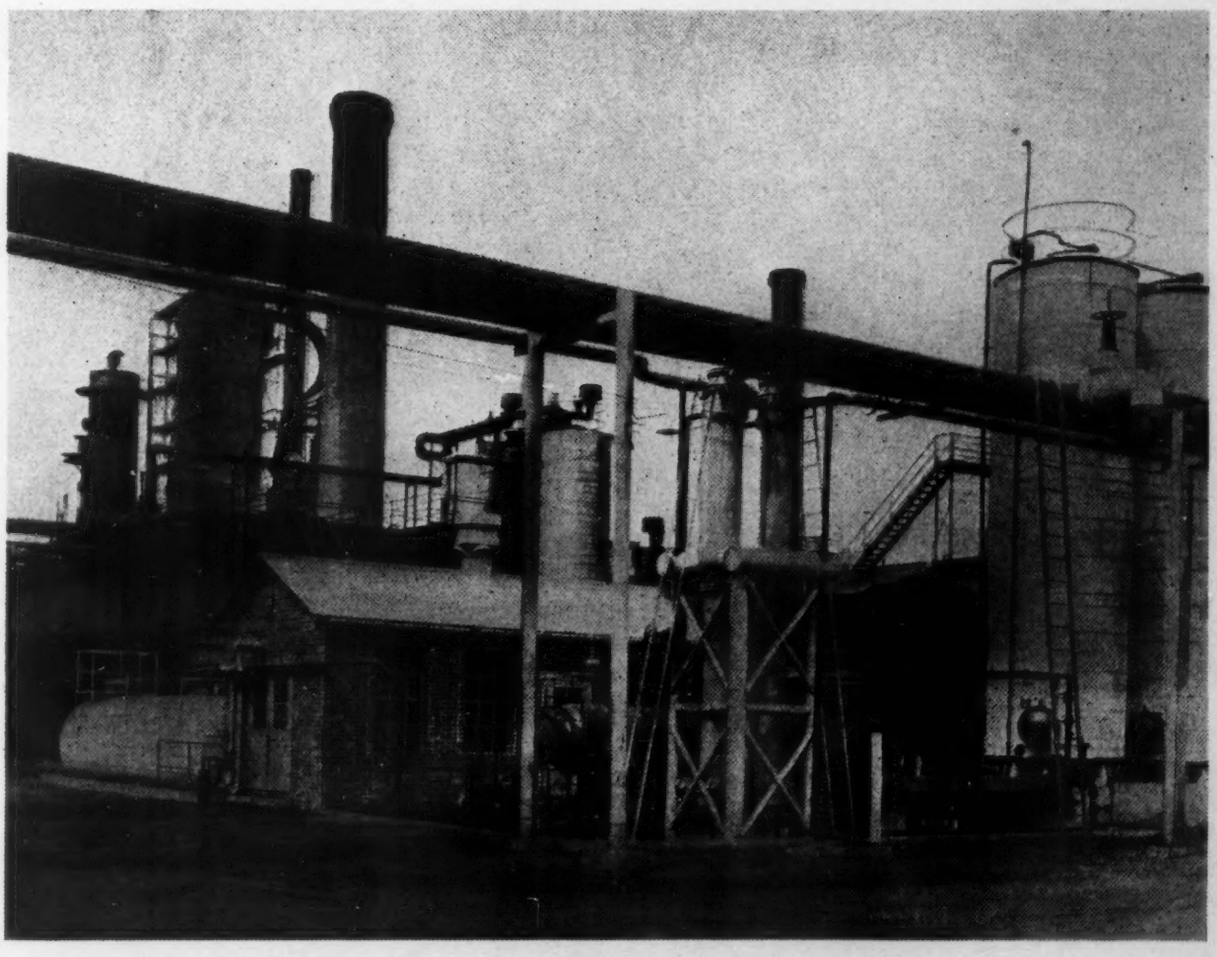

FIGURE 4
Wood River Plant

tively small out-of-pocket expense through the use of idle or abandoned equipment. Through this latter means two plants have been acquired, having a combined capacity of 1300 barrels per day, at an out-of-pocket expense of $35,000 to $40,000 each. This included construction of buildings, contacting equipment-control instruments and some tankage. Principal items of old equipment used included some tankage and distillation equipment.

It should also be noted that no royalty is to be added to the items of cost tabulated above because, according to the present arrangement with the manufacturer of Chlorex,* this item is included in the solvent cost.

* Carbide and Carbon Chemicals Corporation.

VIII. ACKNOWLEDGMENT

The authors wish to acknowledge the cooperation of Carbide and Carbon Chemicals Corporation in developing a licensing arrangement, and of Mid-Continent Petroleum Corporation in supplying some of the information contained herein.

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Solvent Extraction and Its Application

(*Continued from page* 419)

ture-control coils to mixtures 1 and 2, respectively. Each of these mixtures, after effecting intimate mixing between such materials as are admitted to them, discharges the mixture continuously to the corresponding centrifuge which, by the well-known principles of centrifugal force, separates its charge into two homogeneous phases. From centrifuge 1 the solvent phase or spent solvent is discharged to heater **S**, while the oil phase, thus partially refined, passes to mixture 2. Here it is contacted with fresh solvent; and the mixture, as before, is discharged to centrifuge 2. Again, the centrifuge separates its charge into two phases. The oil phase now completely refined goes to heater **O**, and the solvent phase or partially-spent solvent discharges to mixer 1, where its remaining extraction power is utilized. By such counter-current flow there result considerable economies in solvent quantities as well as in recovery of the available paraffinic bodies. By the use of centrifuge, instead of settling tanks, phase separation is greatly speeded up—proportionately shortening the cycle for solvent re-use. In this manner it was found in a plant designed by Foster Wheeler only a few barrels of solvent (exclusive of stand-by storage) were necessary for each 120 barrels of raw stock handled per 24 hours.

From the heaters the two phases pass to their respective vaporizers; from which the vaporized solvent passes to condensers, from which the solvent is returned for re-use. The residual material from the two vaporizers comprises the finished products which pass, either directly or through coolers, to finished oil storage.

It will be evident from the description of the operation that all of the plant equipment is more or less standard and broadly used in the industry, and that no difficulties are presented in the selection of materials for construction.

SUMMARY

In this paper we have tried to give a brief, but comprehensive, idea of the possibilities of solvent extraction.

To summarize, with true solvent extraction it is possible to produce, from comparatively low-grade stocks, oils of low specific gravity, high viscosity index, and lowered carbon residues, and which in the case of neutral oils will not require subsequent acid or clay treatment to meet commercial color specifications. Such a process, being merely a separatory method, will not produce waste products such as sludge and, therefore, will not destroy material. The use of the Foster Wheeler process of solvent extraction will often enable a refiner to produce and utilize in some form products equal to 100 percent of his original material.

It is probable in the near future this method of utilization of organic solvents for petroleum refining will become one of the most important methods of refining and, for that reason has already become of paramount interest to the petroleum refiner.

DUO-SOL Process *In Manufacture of* Lubricating Oils

MALCOLM TUTTLE and
MAX B. MILLER
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A DISCUSSION of the treatment of oil by solvents is of little value unless the operation is considered in relation to the other processing steps required for producing finished motor oils. It is likewise necessary to consider the effect of the solvent process on yield and characteristics of the finished lubricating oils.

DUO-SOL PROCESS SIMPLE AND EFFICIENT

In considering the Duo-Sol process along this line, it becomes apparent that it has unique advantages in both the simplicity of refining operations and in the increased yield of high-quality finished oils. These advantages are obtained in the Duo-Sol process through the application of a new method of solvent extraction. Instead of employing a single solvent, or solvent mixtures, which are selective of naphthenic oils, two solvents are employed—one of which is selective for the paraffinic fraction of the oil charged and the other selective for the naphthenic fraction. Solvents are employed which have a limited solubility in each other, so that the solvents in themselves form a two-layer solvent system.

Utilizing a double-solvent system necessitates the installation of separate recovery units for each solvent. This adds to the initial cost of the extraction plant employing Duo-Sol process; and, based on the oil charged, the unit operating cost is perhaps greater than in those cases where a single solvent is employed. However, the Duo-Sol process goes further in effecting a complete separation than is possible in a single-solvent system, so that higher yields of lubricants of the finest characteristics are obtained. Moreover, it permits the use of residual oils as charging stocks from which paraffinic oils are produced, ranging in color from 4 to 7 N.P.A.—oils which require no acid treatment whatever and which are brought to color at minimum cost. In consequence, the Duo-Sol process should not be considered alone on the basis of its operation, but, on the contrary, should be considered as a part of the entire scheme of producing lubricating oil—inclusive even of the distillation of the crude. This would seem to be particularly true if one is to take the position that over the long pull no extraction process can be justified because of the excellence of its product alone, but must in addition yield all the paraffinic stock in the crude and effect savings in the cost of producing lubricants.

The only crude fraction which contains all of the paraffinic lubricating stock is the residual oil having a flash of approximately 450° F. The yield of the paraffinic oil from such a fraction is governed by the characteristics of the crude and the solvent process employed. The nature of the crude affects yield, in that treatment by solvents is only capable of physically separating the stock into its paraffinic and naphthenic components, and no more paraffinic oil can be produced than is contained in the crude. The solvent process employed affects the yield, in that the degree of separation varies widely with different processes and to the extent to which separation may be carried, in affecting yield, quality, and economy, measures the value of the process.

THE data given in this paper have been the result of the continuous operation of a semi-commercial pilot plant built for the purpose of demonstrating the Duo-Sol process, which has been in continuous operation during the past eight months. The pilot plant was designed and erected in complete detail. It not only incorporates the equipment necessary for the extraction operation, but also employs the apparatus necessary for the continuous recovery of the solvents utilized. It has served both for the purpose of determining the results that could be secured from a large variety of charging stocks, and also as a guide in working out the details of the design of a number of commercial plants employing the Duo-Sol process which are now in process of erection.

This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933.

DUO-SOL ADVANTAGEOUS FOR UNTREATED RESIDUALS

The ability of the Duo-Sol process readily to handle untreated residual stocks is responsible for many of the advantages which may be attributed to the process. Residual stocks, if not used for lubricant production, have only a fuel oil or pressure still charging stock value, which is no greater than that of the naphthenic fraction, if as great. By separating the low value residual stock, without prior treatment, into paraffinic and naphthenic fractions, the refiner avoids processing costs on that fraction which goes into naphthenic oil, and such subsequent refining steps as are required involve only the paraffinic fraction in which the real value lies. In consequence, the ability of the Duo-Sol

process readily to handle residual stocks permits the refiner not only to utilize the cheapest charging stock available from any crude, but also permits the separation to be effected without the depreciation of the value of the charging stock in either of its constituents.

It is not to be thought that the Duo-Sol process will not effectively handle oils which have been partially refined, such as overhead stocks, stocks from which asphaltenes have been removed or, for that matter, which may have been dewaxed. However, it is felt that since each prior refining step adds cost to the value of the stock to be charged to the extraction plant, and by a like amount adds to the cost of the napthenic constituents separated therefrom, greater economy is to be had from the Duo-Sol process as applied to the treatment of the residual materials.

A second advantage to be attributed to the ability of the Duo-Sol process readily to handle residuums is reflected in the yield of paraffinic oil procurable from any charging stock. As has been previously intimated, no extraction process is responsible for the creation of paraffinic oil. It merely separates the paraffinic oil contained in the charging stock to some degree or another. In consequence, the cost of operating any extraction process based on the finished oil will be lowered as the percentage of paraffinic oil produced is increased; or, to put it another way, the cheapest extraction operation is possible when the crude fraction containing the highest percentage of paraffinic oil is processed.

A number of tests have clearly indicated that residual oils, even in the case of crude containing a high percentage of asphalt, will yield a greater percentage of paraffinic oil than will an overhead fraction from the same crude which has been cut to the same initial boiling point. In consequence, it would appear that the ability of the Duo-Sol process readily to handle residuums permits it to utilize a charging stock from any given crude from which the highest yield and, in consequence, the greatest economy is obtainable.

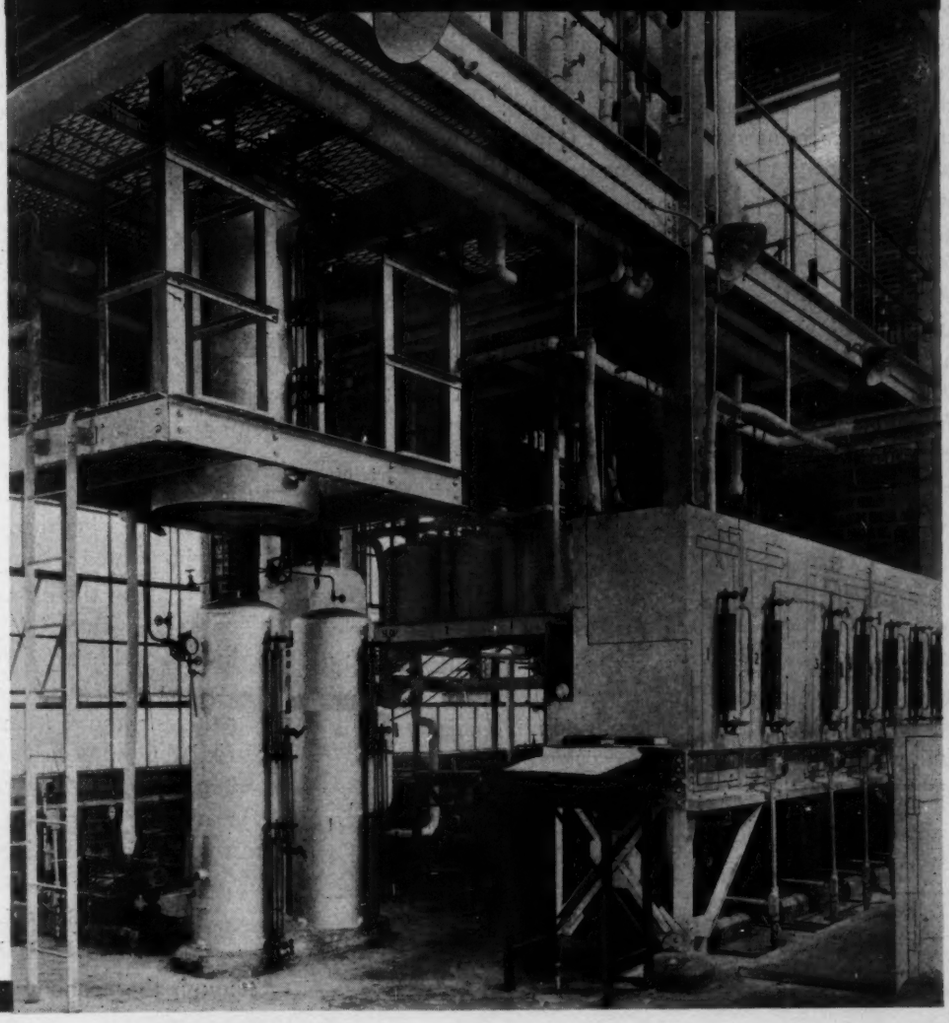
DUO-SOL PERMITS HIGH YIELD OF FINISHED LUBRICANT

Not only does the ability of the Duo-Sol process to handle residuum permit it to utilize the charging stock from which the greatest yield is obtainable, but to a greater extent it also permits the highest yield of finished lubricant in relation to the crude itself. This point would seem to be of exceptional importance. Since they yield in any event is governed by the very nature of the crude, and since the greatest economy is secured from the greatest yield, it would appear that in a great many instances—if not in all—it would be to the advantage of the refiner to select those crudes, of which there are many available, which would be most suitable to the extraction process. If this is done, it then behooves the refiner to procure the maximum yield from the crude run. Also it would seem to be important that he should refine in such a manner as to produce a finished paraffinic oil having the mean viscosity of all grades determined in accordance with the percentage of each grade marketed. If this is done, whether he charges a residual product or an overhead product, the viscosity of the finished product would be identical. If an overhead product is used for the production of an oil having the desired mean viscosity, a comparatively narrow cut must be made which represents a small yield with relation to the crude, as compared with a residual oil yielding a finished product of the identical viscosity. As a matter of fact, tests indicate that the yield, with relation to the crude, of paraffinic oil of the desired viscosity is, in the case of residuums, from two to three times greater than that from overhead stocks; or, to put it another way, if the operator is to charge an overhead stock instead of a residuum and produce therefrom the oil that is required, he would have to run from two to three times as much crude for the same yield of lubricating oil value. If, therefore, it is to the advantage of the refiner to operate his extraction plant at the minimum cost and to select a crude from which the maximum yield can be secured, it is of the utmost importance that a residuum be charged to the plant in order that the highest yield with relation to both the charging stock and the crude can be obtained.

DUO-SOL REDUCES FRACTIONATING AND BLENDING COSTS

There is still a further advantage resulting from the ability of the Duo-Sol process to handle residual oils. It is possible to charge a residual oil to the Duo-Sol process, to dewax the paraffinic oil produced therefrom, to bring the oil to color by filtering, and to secure as a result a base stock which, upon subsequent fractionating and re-blending, will produce a full line of motor oils without an excess or a deficiency of either the heavy bright stocks or the neutrals. As a matter of fact, this ability of the Duo-Sol process curtails to a considerable degree the cost of fractionating and blending for the production of the various grades required. A study of the requirements of several concerns who are distributing motor oils nationally indicates that base stock, ranging in viscosity from 70 to 74 at 210° F., will yield the various grades of oil as required. Such an oil will meet the requirements of S.A.E. 40. Forty percent of the consumption of motor oils in this country calls for such a product, so that 40 percent of such production needs no further refining. Thirty to 35 percent of the consumption requires an S.A.E. 30 oil. Light oil added to the 40 oil will fulfill the 30 oil requirements. In consequence, by extracting a residuum so as to produce an S.A.E. 40 oil only from 25 percent to 30 percent of the total oil produced has to be fractionated into light oil and bright stock. The blending, then, of the two fractions, resulting from this distillation, with the 40 oil affords the refiner the opportunity of manufacturing his full line with a minimum amount of distillation and blending

Interior of Duo-Sol Pilot Plant Showing Solvent Recovery Equipment



Interior of Duo-Sol Pilot Plant Showing Extractor Units in the Foreground.

TABLE 1

Charge Stock	Gravity of Charge (Deg. A.P.I.)	Gravity of Paraffinic Oil Produced (Deg. A.P.I.)	Specific Gravity of Naphthenic Tar Produced	Yield of Paraffinic Oil, Percent with Relation to Charge, before Dewaxing (Percent)	Color of Paraffinic Oil (N.P.A.)	Viscosity of Dewaxed Oil At: 210 Deg. F.	100 Deg. F.	Viscosity Index
Ranger-Burbank residuum	23.5	29.9	1.045	78.4	5½	65	509	100
Tonkawa-Marshall residuum	22.3	29.5	1.047	75.5	6	69	585	100
Oklahoma City residuum	23.9	29.6	1.035	79.5	7+	70	603	100
Hendricks residuum	16.8	27.6	1.017	49.3	4½	67	550	100

when compared with the conventional practices that have been followed heretofore.

CURTAILS FILTERING COSTS

So much for the advantages to be derived through the ability of the Duo-Sol process readily to handle residuums. Earlier in this paper the statement has been made that the Duo-Sol process eliminates the necessity of acid treating, and produces direct from the charging stock paraffinic oils ranging in color from 4 to 7 on the N.P.A. scale. This probably represents one of the outstanding features of the process. It is not only that the cost of acid treating, the cost of acid sludge disposal, the cost of subsequent neutralization, and all of the other inconveniences coincident to acid treating are eliminated; but, in addition, the cost of filtering the oil to final color is materially reduced. An indication as to what may be expected in curtailing filtering costs may be had from the following data.: A long residuum made from the reduction of Ranger-Burbank crude was treated by the Duo-Sol process. The extracted oil was dewaxed by the Sharples process. The dewaxed extracted oil had a viscosity index of 100, viscosity of 66 at 210° F., and a color of 5½ on the N.P.A. scale. A portion of the oil was then percolated through Attapulgus clay which had been tempered at 600° F., so as to make 3, 4, and 5 color on the N.P.A. scale. The yield in each case in barrels per ton is tabulated as follows:

Color (N.P.A.)	Yield (Barrels of Filtered Oil Per Ton of Clay Used)
5	55
4	25
3	8

A second portion of the same oil was contacted with acid-activated clay. The amount of the clay used, per barrel of oil treated, in making similar colors was as follows:

Color (N.P.A.)	Pounds of Acid-activated Clay Used Per Barrel of Oil Contacted (Lb.)
5	10
4	18
3	31

There is a further saving in the cost of filtering the oils to final color. Heretofore it has been necessary to acid-treat and filter oils to color before dewaxing. Duo-Sol extracted oils may be dewaxed as discharged from the Duo-Sol process without rise in pour point upon subsequent filtration. In consequence, the cost of acid treating and of filtering the wax content of the oil is a waste which is no longer necessary.

There is still a further advantage to be gained through the ability of the process to eliminate acid treating. As is well known, the paraffinic oil produced is equally as paraffinic as the oils produced from Pennsylvania crude. In consequence, the oil responds to acid treating in the same manner as do the oils made from Pennsylvania crude. As in the case of Pennsylvania stock, the acid treating of solvent-extracted paraffinic oil is a particularly difficult problem. Following the ordinary procedure of acid treating, it is almost impossible to get a break. Such sludge as is formed seems to stay suspended in the oil. In no case does it seem to be possible to get a break that is complete. In all cases acids of a high degree of concentration are required. In fact, the difficulties of acid treating paraffinic oil, coupled with the cost of bringing such acid oils to final color, lead one to believe that if a stock is to be solvent-extracted at all, it should be refined by this operation to such a degree as to eliminate acid treating entirely. In fact, an analysis has been made which shows that an oil fully refined so as to have a viscosity index of at least 100 can be more cheaply manufactured by the Duo-Sol process than can an oil having a viscosity index of from 85 to 90, simply because of the reduced cost of bringing the highly-refined oil to final color.

Summarizing the above, it is felt that because of the ability of the Duo-Sol process readily to handle raw residuums, and because of the elimination of acid treatment and the reduction in the cost of bringing the oil to final color, the extremes to which it goes are not only justified, but they are responsible—irrespective of the excellent characteristics of the paraffinic oil produced—for an actual saving in the cost of producing oil when compared with prevailing practices.

PRODUCES COLOR STABILITY AND DEMULSIBILITY

Though the economy to be derived from the opera-

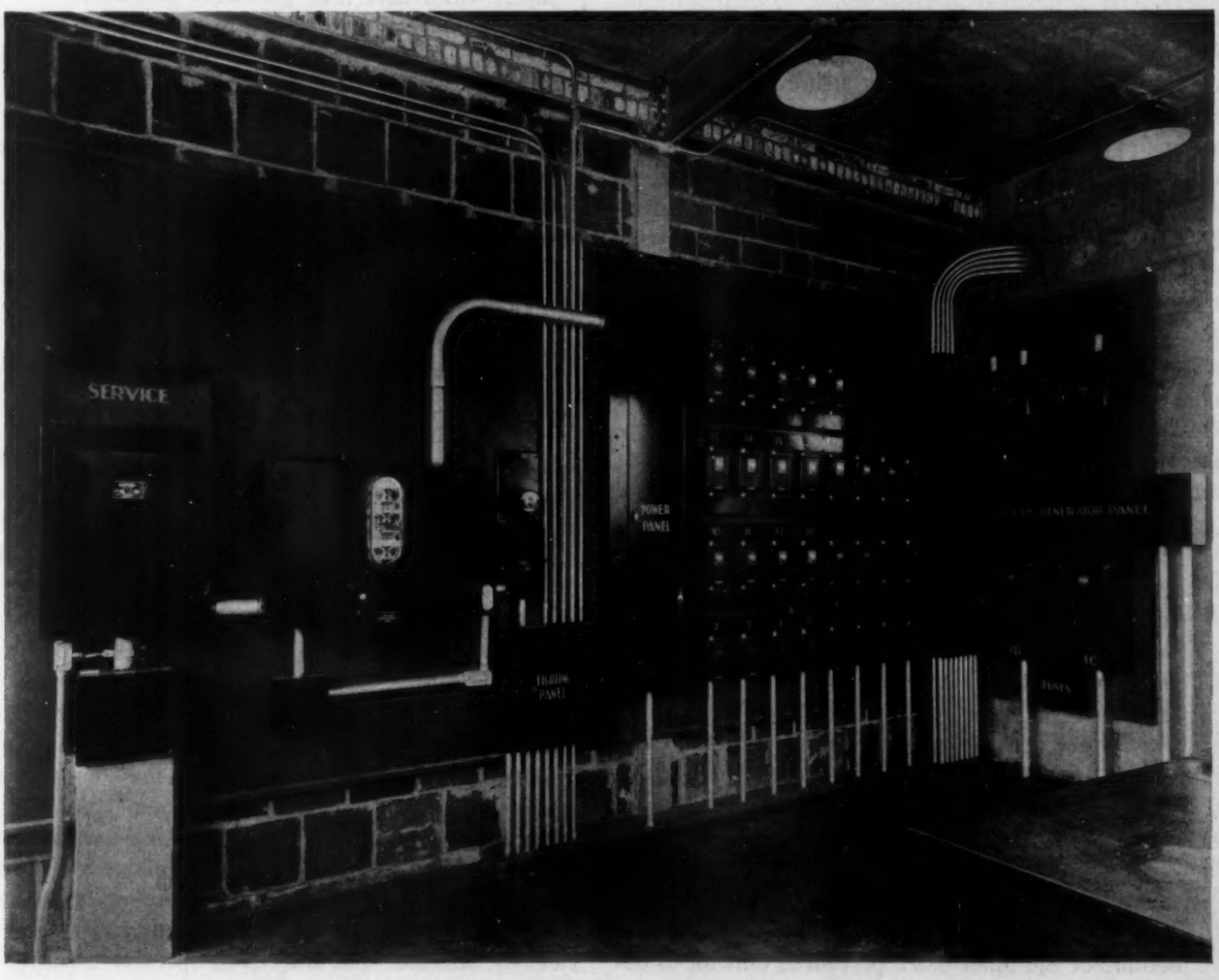
tion of the Duo-Sol process is felt to be of primary importance, a word may be set forth here with regard to the characteristics of the paraffinic oil produced and also the yield of oil produced with relation to the stock charged. As is generally known, the solvent-extracted oils are particularly resistant to oxidation. They are extremely low in carbon residue. Their characteristics in these two respects are measured largely by the degree of separation obtained in the solvent treatment. Oils that have been extracted by the Duo-Sol process, when filtered to final color, are unusually stable as to color, and also have excellent demulsibility —these characteristics being attributed to the fact that no acid has been added to the oil, eliminating the necessity of chemical neutralization.

The yields of paraffinic oil produced from a number of stocks which have been treated by the Duo-Sol process, and the accompanying characteristics of the paraffinic oil produced in each case, are given in Table 1.

A further indication as to the completeness of the separation effected by the Duo-Sol process may be had from a study of the characteristics of the naphthenic extract separated from the charge. For this purpose a sample of the naphthenes separated from the Ranger-Burbank residuum referred to above was reduced by distillation to an asphalt having a penetration of 50. As a result of this distillation 6½ per cent of the naphthenes were taken overhead, the characteristics of which were determined and may be tabulated as follows:

Specific gravity	1.042
Flash point, deg. F., (Cleveland open cup)	250
Viscosity, Saybolt Universal, at 130 deg. F., sec.	257
Viscosity, Saybolt Universal, at 210 deg. F., sec.	51
Viscosity index	Below—50

In recording the data above, the results obtained in the treatment of the first three stocks mentioned were selected for the reason that these stocks are indicative of types of the crude which are particularly suitable for economical Duo-Sol operation. In the case of the Hendricks residuum, however, we have a stock made from a crude which is now being run to asphalt, and from which lubricants of naphthenic characteristics are made from overhead distillate. In consequence, the type of the first three mentioned stocks and the Hendricks residuum represent the extremes in charging stock which may be treated by the process.



Panelboard for Remote Control Electrical Apparatus in Connection with the Duo-Sol Pilot Plant

Use of Phenol *as a Selective Solvent in the Production of High-Grade* Lubricating Oils

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I. GENERAL OUTLINE OF PHENOL-TREATMENT PROCESS

OPERATION OF SARNIA PLANT

IN the operation of the plant pre-heated oil and anhydrous phenol pass counter-currently through a treating system consisting of seven Leaver mixers and seven settling drums with necessary attendant equipment. Treated oil is withdrawn from the top of the last settling drum while spent phenol is discharged from the first settling drum. The spent phenol is cooled, and a light-cycle oil is separated which is returned to the treating system. The treated oil, or raffinate, is pumped to a pipe still where 8 to 15 percent phenol is taken overhead and returned to pure-phenol storage. The bottoms from the tower then flow to a vacuum stripper for final purification from phenol. Analyses indicate that the dephenolized raffinate contains no more than 0.005 percent phenol, which is negligible. The spent phenol contains 25 to 30 percent phenol, which is freed from the extracted oil in a similar manner, and is returned to the process for further use. After purification the extract contains less than 0.07 percent phenol.

This plant has been in service for three years. During this time both very light and very heavy Colombian distillates have been treated without trouble. The only difficulties encountered, which so far have not been wholly corrected, are of a minor nature and are:

a. Fairly-rapid corrosion in the flash coil used for distilling the phenol from the extract: This corrosion is caused by the action of hydrogen sulfide and organic acids, both occurring naturally in the charging stock, and which by the action of phenol are concentrated in the extract. It is hoped that the new re-boilers and dehydrating equipment which have been installed at Sarnia will materially reduce this corrosion.

b. Loss of phenol: The total loss of phenol, as determined by careful inventory, amounted to 0.15 percent by volume during the last three years (based on the charging stock), only half of which can be accounted for in the raffinate or extract. What has become of the other 0.075 percent is difficult to determine. Cartainly part of it is accounted for by mechanical losses, but an explanation of this kind is not satisfactory. It is encouraging to note, however, that during the last three months the unknown loss has dropped from 0.075 percent to 0.04 percent; and it is hoped that with the use of reboilers, which are making a large reduction in the known loss of phenol, the unknown loss can still further be reduced.

Apart from the above difficulties, phenol treating has presented no problems that have not been easily solved.

INTRODUCTION

THE first commercial plant for the phenol treatment of lubricating oils was placed in operation at Sarnia, Ontario, three years ago. This plant has a charging capacity of 2000 barrels per day, and has produced over 30,000,000 gallons of finished lubricating oils, which have been marketed. These oils have given excellent results in all kinds of service, both automotive and industrial, throughout Canada during the past three years. Detailed information concerning this plant has been given in two papers which have been previously published.[1] [2]

It will be of interest at this point to review briefly some of the more important information in these articles in order to provide a background for the new material with which the present paper is primarily concerned.

This paper was presented at the Fourteenth Annual Meeting, American Petroleum Institute, at Chicago, Illinois, October 25, 1933.

OPERATING FACTORS INFLUENCING PHENOL TREATING

There are three important operating factors which have a direct bearing on the yield and quality of the raffinate:

1. *Treating temperature*: The treating temperature is necessarily confined within the limits of the miscibility temperature of phenol and oil, on the one hand, and the solidification temperature of the mixture, on the other; and is ordinarily in the range between 100° and 180°F., although temperatures up to about 250°F. may be used in certain cases. An increase in temperature gives a greater degree of improvement, but lowers the yield of raffinite. The desired temperatures can be attained with the expenditure of comparatively little energy; and, in distinction to other solvent-extraction processes which use low temperatures, phenol requires

no refrigeration and offers no difficulties in the handling of waxy stocks.

2. *Proportion of oil to phenol*: An increase in the proportion of oil to phenol has an effect somewhat similar to that obtained with an increase in temperature. The optimum treating conditions, as far as these two factors are concerned, are largely governed by the properties of the distillate to be treated and the improvement desired. In general, 100 to 175 percent phenol by volume is used.

3. *Contacting of phenol and oil:* The contacting of phenol and oil may be carried out by a batch method or counter-current treatment. Obviously, the latter is more advantageous, and is used in commercial practice.

STOCKS WHICH HAVE BEEN TREATED

The operation of the commercial plant at Sarnia has been confined to lubricating distillates from Colombian crude, since these fractions have low pour points and require no dewaxing, except in the heavier motor oil grades from which a slight wax haze must be removed by centrifuging. A wide variety of fractions has been handled successfully, ranging from low-viscosity cuts for the production of transformer and turbine oils to distillates of 150 Saybolt viscosity at 210°F. for the heavier motor oil and aviation grades.

In addition to Colombian oils, a great deal of work has been done on cuts from other crudes in laboratory-scale apparatus. Over 100 stocks have been treated, including fractions from Mid-Continent, Reagan, Ranger, Van Zandt, East Texas, West Texas, Santa Fe Springs, Coastal, Sugarland, Pecos, Pennsylvania, and other crudes. Many of these stocks have been treated in their waxy condition. Results on some of these stocks are presented in the latter part of this paper, together with data on additional stocks recently treated.

GENERAL RESULTS OF PHENOL TREATMENT

The results obtained from the phenol extraction of a lubricating fraction may be summarized as follows:

1. *Viscosity index*: Operation of the Sarnia plant has shown consistent improvements of 45 to 55 points in viscosity index with Colombian stocks. Greater improvement could be obtained with a sacrifice in yield. Stocks of higher initial viscosity index have been treated to 95 to 100 viscosity index and above, with good yields in the laboratory.

2. *Yield*: This factor is, in general, a function of the improvement in viscosity index between charge stock and raffinate and, to a lesser extent, a function of the viscosity index of the feed stock. A plot is shown in one of the papers previously mentioned[2] which presents this relationship. In the Sarnia plant raffinates of 75 to 85 viscosity index have been obtained from Colombian oil with yields of 55 to 65 percent.

3. *Conradson carbon reduction*: In general, the Conradson carbon of the lubricating oil is reduced from 60 to 95 percent by phenol treatment. This is illustrated in the accompanying tables which will be discussed hereafter.

4. *Flash point*: Phenol-treated oils possess, in general, the same flash points as the stock from which they are prepared. If oils of high flash are treated, raffinates of high flash will be obtained.

5. *Gravity and color*: A marked improvement is noticeable in these two characteristics after phenol treatment. Reference to the tables included in this paper will illustrate the magnitude of these changes.

6. *Reduction of Sligh oxidation number*: In addition to the above, phenol extraction markedly improves the stability of the oil towards oxidation, as measured by Sligh oxidation number. Tests on a variety of stocks have shown 93 to 99 percent reduction. For example, a heavy Colombian stock of 146 Saybolt viscosity at 210°F. before treatment showed a Sligh number of 87.5, and after phenol extraction a value of 0.8. The same stock with acid treatment and clay contacting was reduced to 4.5 Sligh number, indicating the superiority of phenol over acid and clay.

7. *Sulfur reduction*: The sulfur content is similarly reduced. In the case of Colombian distillates this amounted to 60 to 78 percent, depending on operating conditions.

8. *Steam-emulsification number*: Low steam-emulsification numbers are obtained, particularly for the heavy-motor oil grades.

FINISHING OF PHENOL-TREATED OILS

It has been found in the operation of the Sarnia plant that finished motor oils can be produced from the phenol raffinate without the necessity of any other finishing process than clay contacting. In the actual plant processing at the Sarnia refinery, acid finishing of lubricating oils of all descriptions has been largely eliminated by use of phenol treatment. Summer and winter-grade aviation oils produced by phenol treatment, followed by simple clay contacting, have met specifications of the British Air Ministry, which include very severe oxidation and coke tests.

The various S.A.E. grades are produced at Sarnia by phenol treating flash-coil distillates of the proper viscosity; for this reason all the motor oils are narrow-cut distillates.

Since Colombian crude is used, dewaxing has been unnecessary except for removal of a slight wax haze in S.A.E. 40, 50 and 60 grades. In handling waxy feed stocks it would, of course, be necessary to dewax. This would preferably be conducted after phenol treatment in order to reduce the quantity to be dewaxed and to avoid a slight rise in pour point which may take place during phenol extraction. It is evident that the small amount of finishing required for phenol-treated distillates is an important advantage for the process.

UTILIZATION OF EXTRACTED OIL

Because of its aromatic nature, the extract yields high-octane-number gasoline on cracking, and may also

be oxidized to yield asphalt. In the case of charging stocks of low viscosity, the extract may be worked up by any of the well-known methods to yield low-viscosity-index lubricating stocks.

II. ENGINE TESTS AND NEW PHENOL-TREATING DATA

Having outlined the general nature of the phenol-treatment process, two phases of the process which have not been stressed so far will now be taken up in detail:

1. The excellent results which have been obtained in the laboratory, road, and airplane engine tests using narrow-cut, high-colored phenol-treated oils.
2. The wide application of the phenol process to the treatment of various lubricating stocks. In this connection data will be presented on the results obtained by phenol treating residual stocks, distillates, oils before and after dewaxing, and oils having viscosity indices from —60 to 100.

ENGINE TESTS

In spite of the recent articles appearing in the petroleum journals extolling the use of heavy bright stocks in motor oils, there are few, if any, scientific data substantiating such claims. The work that has been carried on during the last three years in the engine laboratory of Standard Oil Development Company, in conjunction with tests on phenol-treated Colombian distillates, indicates the superiority of narrow-cut highly-refined oils. These same results have been obtained in general practice with phenol-treated oils throughout Canada and with phenol-treated airplane oils in Europe.

This was demonstrated as early as May, 1930, by the engine laboratory mentioned above in a 50-hour test using a four-cylinder White truck engine. Nine commercial oils with viscosity indices up to 100, and three narrow-cut, highly-treated oils, all having viscosities of approximately 75 sec. at 210°F., were tested. In all the runs, made under very carefully-controlled conditions, the three narrow-cut oils gave by far the best results, with the phenol-treated oil ranking exceptionally high.

While it was better in every respect as compared with the nine commercial oils, the consumption of oil was somewhat higher than with the other two narrow-cut oils. This was explained in part by the fact that the phenol-treated oil had a slightly lower viscosity at 210°F.

In another series of motor tests with a Mack "B.G." truck engine, in December, 1930, six commercial and three especially-prepared oils of approximately 62 viscosity at 210°F. were used. As before, the phenol-treated narrow-cut, light-colored oil ranked with the best and well above the commercial oils.

In subsequent work a number of test runs were made on a Chevrolet six-cylinder engine. All the oils tested were of the S.A.E. 40 grade, and included a hydrogenated oil, a phenol-treated oil, and a narrow-cut, highly-refined, acid-treated distillate. The tests indicated all three oils to be very high quality, with the hydrogenated and the phenol-treated oils as good or superior in every respect to any other oil on the market.

In contrast to these very favorable results, phenol-treated Marvelube "A-5" caused some ring sticking, in recent 15-hour tests with a C.F.R. engine in the Development Company's laboratory, when various aviation oils were tested for that defect. However, phenol-treated oils have been used successfully during the last three years in airplanes both in Canada and Europe, indicating that this test is probably too severe. These satisfactory results are further borne out by a recent test run made in the Wright Aeronautical Laboratory using this type of oil in a Cyclone engine. The results of a 50-hour test were excellent in every way, with no sign of ring sticking or gum formation.

In addition to the above laboratory tests, phenol-treated oils have given excellent results in all kinds of service—both automotive and industrial—throughout Canada during the past three years.

TABLE 1

California Low-Viscosity-Index Distillates

Treatment Inspections	Stocks: 70-Viscosity Untreated Coalinga 125 Percent Phenol at 90 °F.		150-Viscosity Untreated Coalinga 100 Percent Phenol at 100 °F.	
	Before	After	Before	After
Viscosity, at 210°F.	69	59	139	92
Gravity, °A.P.I.	15.9	26.2	14.2	21.3
Viscosity index	—50	+74	—60	+31
Conradson carbon residue	0.215	Nil	0.704	0.103
Pour/solid, °F.	25/20	—5/—10	45/40	5/0
Robinson color	Transparent green	17½	Transparent green	3½
Yield, percent	.....	24.7	.....	36.2

PHENOL-TREATED LUBRICATING OILS FROM VARIOUS CRUDES

Within the last six months some 40 new stocks have been treated, and almost every type and degree of re-

TABLE 2
Coastal and Colombian Distillates

Treatment Inspections	Stocks: 60-Viscosity Coastal 100 Percent Phenol at 115 °F. Before	After	Extra-heavy Colombian 115 Percent Phenol at 125 °F. Before	After
Viscosity, at 210°F.	64	60	146	99
Gravity, °A.P.I.	20	25.8	17.4	25.8
Viscosity index	22	74	28	77
Conradson carbon residue	0.125	0.029	1.845	0.295
Pour/solid, °F.	—10/—15	0/—5	30/25	45/40
Robinson color	Blue opaque	9¼	Green opaque	¾
Yield, percent	.....	64.7	.....	53.3

finement has been represented. The oils that have been treated varied as follows:

1. Very low- and very high-viscosity-index oils.
2. Narrow- and wide-cut oils.
3. Very clean and very dirty stocks.
4. Heavy-residual and light-overhead cuts.
5. Waxy and dewaxed oils.

Work on these stocks has indicated the types of operation to which the phenol plant is best adopted. Among these have been fractions from Mid-Continent, Panhandle, Oklahoma City, Coalinga, Ventura, Coastal, and Pennsylvania crudes.

In the time available all the stocks treated were not handled under optimum conditions, and much of the work is being continued. However, there is enough evidence to permit drawing certain conclusions, which can be best brought out by giving representative examples of the various stocks treated.

It can be seen from Table 1 that a great improvement in gravity, viscosity index, and color has been obtained; and in both cases the Conradson carbon residue has been lowered 85 percent or better. Oddly enough, with these particular oils, the cold test has been lowered by phenol treating. This peculiarity was observed some years ago when Peruvian distillates were given a light phenol treat. All the improvements shown are naturally obtained at the expense of yield and viscosity. Whether or not there is more medium viscosity-index material in the extract still has to be determined.

The lighter-viscosity oil improves in viscosity index to a greater extent than the heavier stock, yet does not drop as much in viscosity. This is characteristic of most of the oils studied.

The color improvement is so great with stocks such as these that little, if any, further treatment would be necessary, except possibly to improve the cast.

Table 2 gives the treatment of Coastal and Colombian distillates. There is nothing unusual in these two treats, except to note that the Coastal oil has the same A.P.I. gravity and viscosity index as the Colombian distillate —although the viscosity is 39 sec. lower. Again, the drop in viscosity is greater for the higher-viscosity oil. Both stocks only require a light clay treatment to insure good color and color stability.

In Table 3 are shown the results of phenol treating Mid-Continent undewaxed stocks. It is a general rule that phenol treating removes almost all the volatile coloring matter from clean distillates. If a stock contains entrained asphaltic material, or if a residual oil is treated with phenol, very little improvement in color is obtained, as is shown to be the case with the Oklahoma residual mixture. A great deal of coloring matter is, of course, removed from the oil, but a very inert black material remains. Small quantities of acid are required to remove this black material from phenol-

TABLE 3
Mid-Continent Undewaxed Stocks

Treatment Inspections	Stocks: 100 Wax Slops 3-100 Percent Batch Treatments at 210 °F. Before	After	Oklahoma City-Crude Mix 150 Percent Phenol at 140 °F. Before	After
Viscosity, at 210°F.	98	78	144½	111
Gravity, °A.P.I.	22.6	28.5	19.8	25.8
Viscosity index	74	107	71	103
Conradson carbon residue	1.330	0.179	5.595	2.866
Pour/solid, °F.	70/65	100+	70/65	70/65
Robinson color	Blue opaque	Red	Black	Greenish black
Yield, percent	.....	65.4	.....	67.8

TABLE 4
Mid-Continent Dewaxed Stocks

Treatment Inspections	Stocks: Untreated 300-Viscosity Neutral 6-50 Percent Batch Treatments at 105 °F. Before	After	100 Bright Stock 150 Percent Phenol at 200 °F. Before	After
Viscosity, at 210°F.	54	50	118	106
Gravity, °A.P.I.	25.2	30.9	24.1	27.3
Viscosity index	86	109	80	96
Conradson carbon residue	0.09	Nil	1.215	0.580
Pour/solid, °F.	—10/—15	0/—5	0/—5	15/10
Robinson color	Blue opaque	10	Pea green	No change
Yield, percent	.....	59.6	.....	72.8

treated oils, as simply contacting with clay makes no appreciable improvement.

The Conradson carbon residue is more difficult to lower with certain residual stocks than with a distillate, although it must be remembered that Conradson carbon is not only produced from easily-decomposed asphaltic material, but also from very high-viscosity oils which may break down under the conditions of the test producing carbon. This explains why the Oklahoma City residuum, which is a wide cut, has not given as great a reduction in carbon residue as many of the other stocks shown.

Table 4 gives the results of phenol treating two dewaxed Mid-Continent stocks. Because these two oils are cleaner than those shown in the previous table, the carbon residue in both cases has been greatly reduced, and at the same time the drop in viscosity at 210°F. is not as great as with the dirtier oils. It will be noted that the pour point is only slightly changed.

The phenol treatment of a Pennsylvania steam-reduced stock is presented in Table 5. Indications are, both from the flash point and the low Conradson carbon of this stock, that it does not contain as large a quantity of high-boiling-point fractions as occur in Mid-Continent residual stocks. This supposition is further borne out by the fact that the Conradson carbon is greatly reduced by phenol treating, and is low for the high viscosity of the oil.

Another interesting point to note, which appears to be peculiar to Pennsylvania stock, is that by successive treatments with phenol the viscosity of the raffinate is first lowered and then increased. In fact, with still heavier treatments by phenol, and consequent sacrifice in yield, it has been possible to raise the viscosity of the raffinate well over that of the original oil and at the same time increase the viscosity index to as high as 121.

SUMMARY AND CONCLUSIONS

1. Treatment with anhydrous phenol offers a means of producing high-grade lubricating oils from a wide variety of stocks. Actual test has demonstrated that stocks of the following types may be handled.
   a. Very low- and very high-viscosity index oils.
   b. Narrow- and wide-cut oils.
   c. Very clean and very dirty stocks.
   d. Heavy-residual and light-overhead cuts.
   e. Waxey and dewaxed oils.

2. The process is applicable not only to the manufacture of motor and aviation oils, but also to turbine, transformer, and other special oils.

3. The commercial feasibility of the process has been demonstrated by three years' successful operation of a 2,000-barrel-per-day plant.

4. The high quality of the phenol-treated oils has been demonstrated not only by laboratory tests, but by three years' service in Canadian automotive and industrial markets.

BIBLIOGRAPHY

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[2] Stratford, Moor, and Pokorny, "Phenol as a Selective Solvent in the Refining of Lubricating Oils," *World Pet. Conf.*, London, July 19-25, 1933.

TABLE 5
Pennsylvania Steam-refined Stock

Treatment Inspections	Stocks: at 200 °F. 75 Percent Phenol Before	After	125 Percent Phenol at 250 °F. After
Viscosity, at 210°F.	230	203	223
Gravity, °A.P.I.	24.7	26.2	28.1
Viscosity index	102	108	113
Conradson carbon residue	3.190	2.120	1.18
Pour/solid, °F.	30/25	50/45	55/50
Robinson color	Brownish Green	Green 91.5	Green 54.6
Yield, percent	.....	91.5	54.6

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Fundamental Physical Data

Pressure-Temperature and Low Pressure Total Heat Relationships of Petroleum Fractions. E. S. RAGATZ, E. R. MCCARTNEY *and* R. E. HAYLETT. *Ind. & Eng. Chem.* **25** (1923) pp. 975-79.

A method of identifying the equivalent pure-cut a wide-cut fraction has been developed. Analysis of available data shows that the critical point for such a cut constitutes the common point of convergence for the dew lines of oil similar wide-cut fractions of equal gravity. This equivalent pure-cut critical point can also be used as a guide for the development of wide-cut boiling lines and partial equilibrium vaporization. Once the equivalent pure-cut has been established, the physical data pertaining to this cut can be used as a basis for calculating the total heat of petroleum fraction vapor existing at low pressures. A dew point still and its method of operation are described. Diagrams showing the procedure and methods of calculation are given as well as a typical total heat diagram. The method is applied to a series of fractions from a Los Angeles basin crude.

Dielectric Constants of Naphtha Products. T. G. KOVALEV *and* V. V. ILLARIONOV. *J. prakt. Chem.* **137** (1933) pp. 257-60.

Dielectric constants are given for benzene, pseudocumene, naphthalene, acenapthene, phenanthrene, hexane and decane. The values found indicate an increase in dielectric constant as each new ring enters the molecule. This increase is not so striking for an increase in the length of the chains. The content of aromatics, naphthenes and aliphatic compounds are given for samples of kerosene, distillate, heavy solar oil distillates, spindle oil, and machine oil.

Rate of Solution of Methane in Quiescent Liquid Hydrocarbons. R. D. POMEROY, W. N. LACEY, N. F. SCUDDER, *and* F. P. STAPP. *Ind. Eng. Chem.* **25** (1933) pp. 1014-19.

Increasing use of the process of repressuring by forcing gases into an oil formation has led to need for more accurate knowledge of the nature of the changes taking place during this process. For example, the extent to which gas will dissolve and diffuse into oil in a formation. The theory of the subject is developed. An apparatus was constructed that allowed the determination of diffusion constants and solubilities of gases in liquids at pressures up to 30 atmospheres. The results obtained using methane and dissolving it in iso-pentane are found to be in good agreement with the theoretical equation. Consideration of the assumption involved, together with the consistency of the results, indicates that the method is capable of giving fairly reliable values for the true absolute diffusion constant. The probable error may be as great as 4 per cent. A study of the extent to which the diffusion constant is affected by pressure or by the concentration of the methane gas in solution up to 20 atmospheres did not reveal any effect.

Chemical Composition and Reactions

Preparation of Tetramethylmethane (Neopentane) and Determination of Its Physical Constants. F. C. WHITMORE *and* G. H. FLEMING. *Jour. Am. Chem. Soc.* **55** (1933) pp. 3803-6.

Methods of preparing tetramethylmethane were studied. The method finally adopted was the action of tertiary butyl chloride and methyl magnesium chloride, both in toluene at 45° to 50° C. Consistent yields of 42-50 per cent were obtained repeatedly. Data are given covering the refractive index, boiling point, freezing point, density, molecular refraction, and vapor pressure.

Isolation of Ethylbenzene from an Oklahoma Petroleum. J. D. WHITE *and* F. W. ROSE, JR. *Bur. Stand. J. Res.* **10** (1933) pp. 639-645.

Ethylbenzene was extracted by means of liquid sulphur dioxide from a petroleum fraction distilling between 130° and 145° C. The material was purified by repeated extraction and distillation, and finally by sulphonation and hydrolysis. The properties of the compound are given.

The Analysis of Hydrocarbon Oils. R. H. GRIFFITH *and* H. HOLLINGS. *Jour. Inst. Pet. Tech.* **19** (1933) pp. 701-712.

Paraffins and naphthenes were isolated from the gas oil fraction of a number of oils. It was found that the portion insoluble in fuming sulfuriq acid corresponds with that least soluble in aniline. The component substances are termed paraffins. The aniline point-boiling point curve for the isolated paraffins and naphthenes are given, and their mutual effect on each other's aniline point determined. It was found that the previous assumption of a lowering of 0.4°C for 1% naphthene is satisfactory. The aniline point-boiling point curves for crude oils are straight lines, whereas crudes that have suffered partial cracking give curves. Data are discussed in relation to the analytical determination of the value of gas oil for gas making.

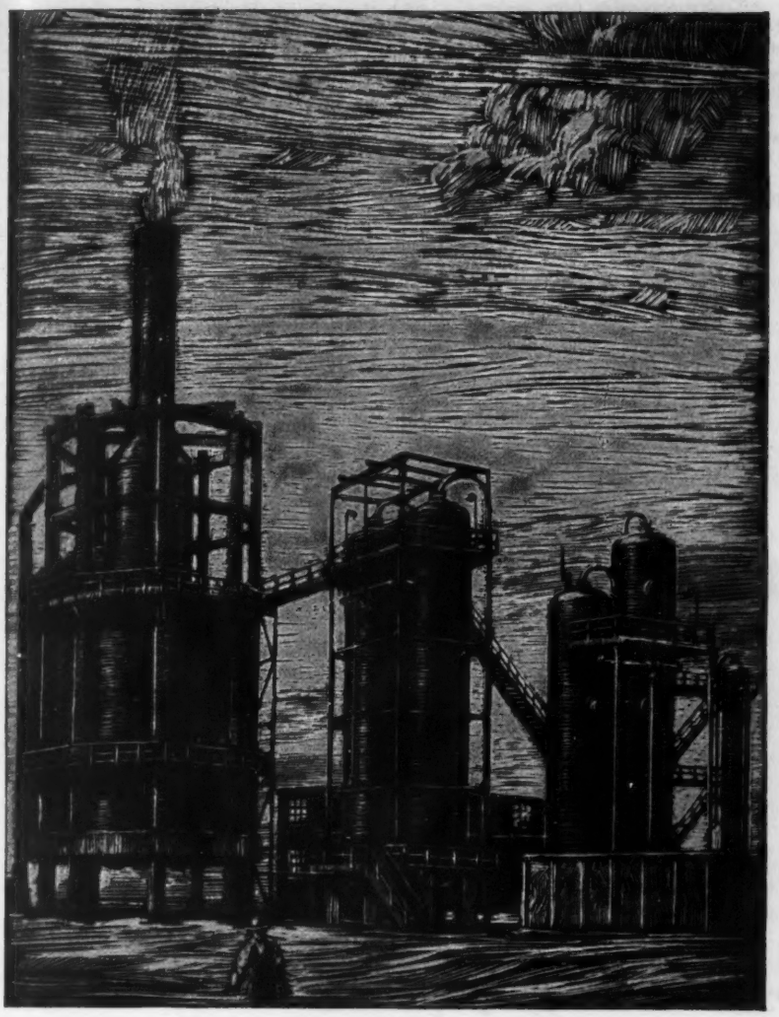
Manufacture: Processes and Plant

Recovery of Hydrogen Sulphide from Petroleum Refinery Gases. L. ROSENSTEIN. *Petroleum* **29** (1933) No. 24, pp. 4-6.

Because of cheapness, high absorptive power and non-volatility tri-potassium phosphate in concentrated solution is one of the most suitable reagents for the absorption and recovery of hydrogen sulphide from refinery and other gases. The maximum absorptive power of the solution at 25° is determined by the solubility of di-potassium hydrogen sulphate at that temperature. The absorptive power is estimated as 55 g. of hydrogen sulphide per 1,000 g. of solution. The largest item of cost in the process is the steam used in the generation of the solution. The estimation of steam consumption is discussed.

Conditions under Which Gas and Liquid Mixtures Rise. J. VERSLUYS. *Jour. Inst. Pet. Tech.* **19** (1933) pp. 694-700.

The paper discusses the principles limiting the conditions of flow of a gas and a liquid through a vertical pipe. When a mixture of liquid and gas rise in a tube two stable conditions are possible, i. e. foam and mist. The foam condition arises when more liquid is present than gas, and the mist condition when more gas than liquid. Diagrams are given illustrating the different possibilities. Between the conditions of stable foam and stable mist is a condition of instability of either type in which flow in slugs occurs. The paper is of



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Removal of Thiophene from Benzene by Catalytic Hydrogenation. R. N. PEASE, W. B. KEIGHTON *and* W. C. MUNRO. *Ind. Eng. Chem.* **25** pp. 1012-13.

Investigations have shown that the sulphur compounds present in petroleum naphthas may be removed by the passing of the vapor along with hydrogen over a nickel catalyst at 300°-400°C. Thiophene proved to be the most resistant of the sulphur compounds. This substance was, therefore, used in further studies of the problem and benzene was substituted for petroleum naphtha as a carrier in order to have more definite conditions, and, also, because thiophene is a common impurity of benzene and is difficulty removable from benzene. Using a cobalt sulfide molybdenum oxide catalyst resulted in substantially quantitative elimination of sulphur and negligible hydrogenation of benzene. Cobalt sulfide and chromium oxide alone were also found to be good catalysts for the removal of thiophene.

Products: Properties and Utilization

The Phenomenon of Knocking. E. S. TAYLOR. *Aircraft Eng.* **5** (1933) pp. 136-7.

Ricardo's theory that knocking is the result of autoignition in the last part of the charge to burn is extended. The self-ignition temperature of normal heptane is reached with a compression ratio of 3.75:1. However, this fuel is used in an engine with this compression ratio without knocking. The explanation is that there is insufficient time for completion of the physical and chemical changes that cause self-ignition before the flame front passes through the mixture and completely consumes it. Curves are presented showing the compression ratios for autoignition for various fuels with and without addition of tetraethyl lead.

Tractor Fuels. E. C. SAUVE. *Mich. Agr. Expt. Sta., Quarterly Bull.* **15** (1933) pp. 287-92.

The most economical fuels for tractor use in order of preference based on performance and cost are distillate, kerosene, gasoline, alcohol-gasoline mixture. Gasoline causes less dilution of the crank case oil than kerosene or distillate, and hence contributes to more efficient lubrication and longer life of engine parts. The use of crude oil in tractors is of questionable economy.

Estimation of Residue-Formation in Motor-Oils. PHILIPPOVICH. *Erdol u. Teer,* **9** (1933) pp. 236-237, 253-254, 267-268.

The relation of the tests of lubricating oils to their behavior in practice is discussed and it is concluded that the most important properties are the viscosity and the tendency to form coke in the cylinder and sludge in the crank case. The mechanism by which these residues are formed is considered. Crank case sludge, or at least the solid portion of it, has approximately the same ultimate composition as the coke in the cylinder, namely 75% carbon, 4% hydrogen, and 21% oxygen. Previous work on carbon formation is reviewed, and a discussion given of the methods of Conradson and Ramsbottom, as well as the oxidation methods of Butkow, Baader, and the English Air Ministry. Six oils of approximately the same viscosity were studied. Evaluation of these oils from their engine performance and formation of coke and sludge placed them in a series from one to six, six being the worst. The oils were then evaluated using the various residue tests. The Conradson test agreed with the engine tests except that oil 3 was worse than oil 4. The Ramsbottom method also showed 3 worse than 4, and 5 slightly worse than 6. The carbonization in air tests gave results similar to those of the Conradson, but 1 and 2 and 4 and 5 were reversed. The results of the Butkow oxidation test were not in agreement with the engine tests. The Air Ministry tests agreed approximately with the engine tests except for oil No. 1 which contained some fatty oil. This showed worse in the test than in practice. It was concluded that further work is needed to develop a simple test the results of which will correlate with the behavior of oils in an engine.

Theory of Film Lubrication. A. M. ROBB. *Proc. Roy. Soc.* **A. 140** (1933) pp. 668-694.

The study of Goodman's experimental results leads to discovery of a flaw in the statement of Reynolds' fundamental theory. $dp/dx\ \lambda = u\ (h - h'/h^3$. The value of h' is always taken as the height at the section of maximum pressure and so is a constant. It can, in fact, be a constant only in the hypothetical case of a bearing of infinite axial length. Therefore, it is necessary to consider the out-flow and in-flow of the lubricant based on these modified conceptions. Agreement is obtained between calculated and experimental results as regards attitude and

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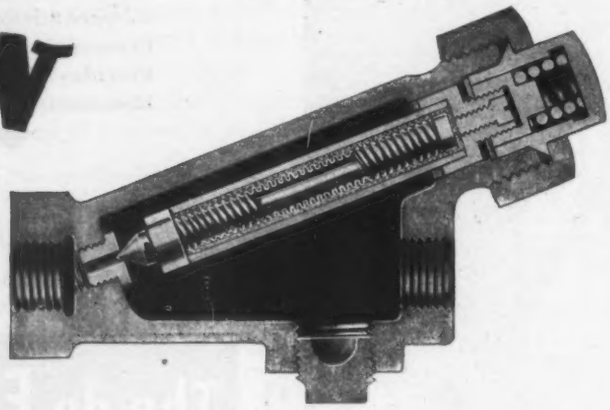

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Mineral-Oil Asphalts. H. POLL. *Petroleum* Z. **28** No. 36, Asphalt u. Strassenbau (1932) pp. 2-7.

Available methods for the separation of asphalt into distinct and chemically unchanged fractions are critically reviewed and experimentally compared. An efficient method is described in which the asphalt is exhaustively extracted with cold light petroleum solvent and the solution shaken with fullers earth which is then separated. Evaporation of the solution gives a mineral oil fraction. The fullers earth is extracted with cold chloroform giving mineral oil resins. The asphaltenes remaining after extraction of the original asphalt are dissolved in chloroform and the solution shaken with fullers earth. The separated fullers earth is extracted with pyridine or carbon disulphide giving a solution of asphalt resins. Mineral oils and asphalts may be separated by this method into the fractions given together with a carbene fraction and an insoluble carboid fraction.

The Performance of High-Speed Diesel Engines for Road Transport as Affected by the Nature of the Fuel. J. G. WITHERS. *Jour. Inst. Pet. Tech* **19** (1933) pp. 713-24.

Many designs of high speed Diesel engines have appeared that operate successfully only on certain fuels. The purpose of the work here reported was to study fuel characteristics in relation to engine behavior. The fuel must be free from foreign matter. The viscosity must be low enough to meet the coolest conditions likely to occur in the pump system and high enough for the oil to be pumped with small leakage and to lubricate the working parts. The carbon residue should be as low as possible to avoid internal carbonization in poorly designed engines. High boiling fractions should not be present if a clean exhaust is desired under all conditions. For silent running, easy starting, absence of shock loading, and good idling, a fuel of short delay or good ignition quality is desirable. As high a specific gravity as possible, consistent with short delay and reasonable distillation range, and as high a calorific value as possible are required to insure maximum power and miles per gallon.

Kinematic Viscosities and Times of Outflow from Commercial Viscometers. F. H. GARNER *and* C. I. KELLY. *Physics* **4** (1933) pp. 97-112.

The statement of viscosity in terms of the units peculiar to the various types of viscometers (Saybolt, Redwood, and Engler) introduces difficulties that would be eliminated by the international acceptance of the c.g.s. units, particularly the "Stoke," as the unit of kinematic viscosity, since this unit does not involve a knowledge of density. The main part of the paper comprises a discussion of the present positions held by the three viscometers.

Automatic Pressure Regulators for Vacuum Distillation. E. B. HERSHBERG *and* E. H. HUNTRESS. *Ind. Eng. Chem., Anal. Ed.* **5** (1933) pp. 344-6.

An automatic pressure regulator for vacuum distillation is described. The manostat contact actuates the grid circuit of a 71-A type vacuum tube which controls the operation of a 2,000 ohm magnetic relay. Regulation of pressure to ±0.015 mm. of mercury is secured at any pressure up to atmospheric.

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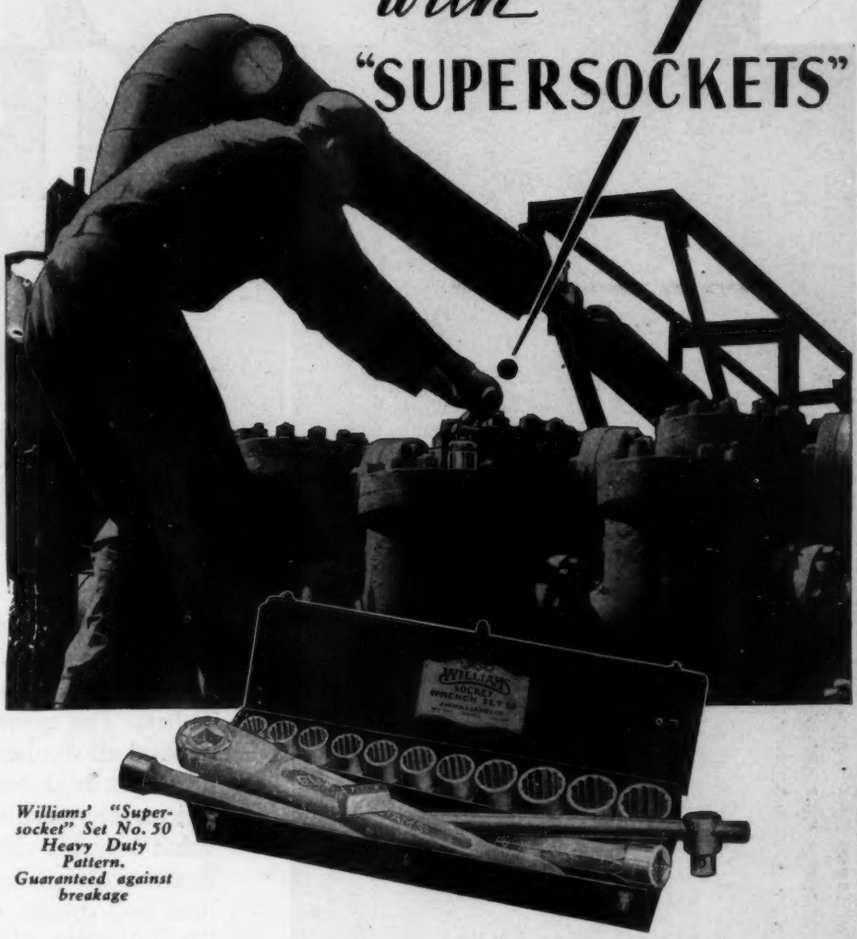

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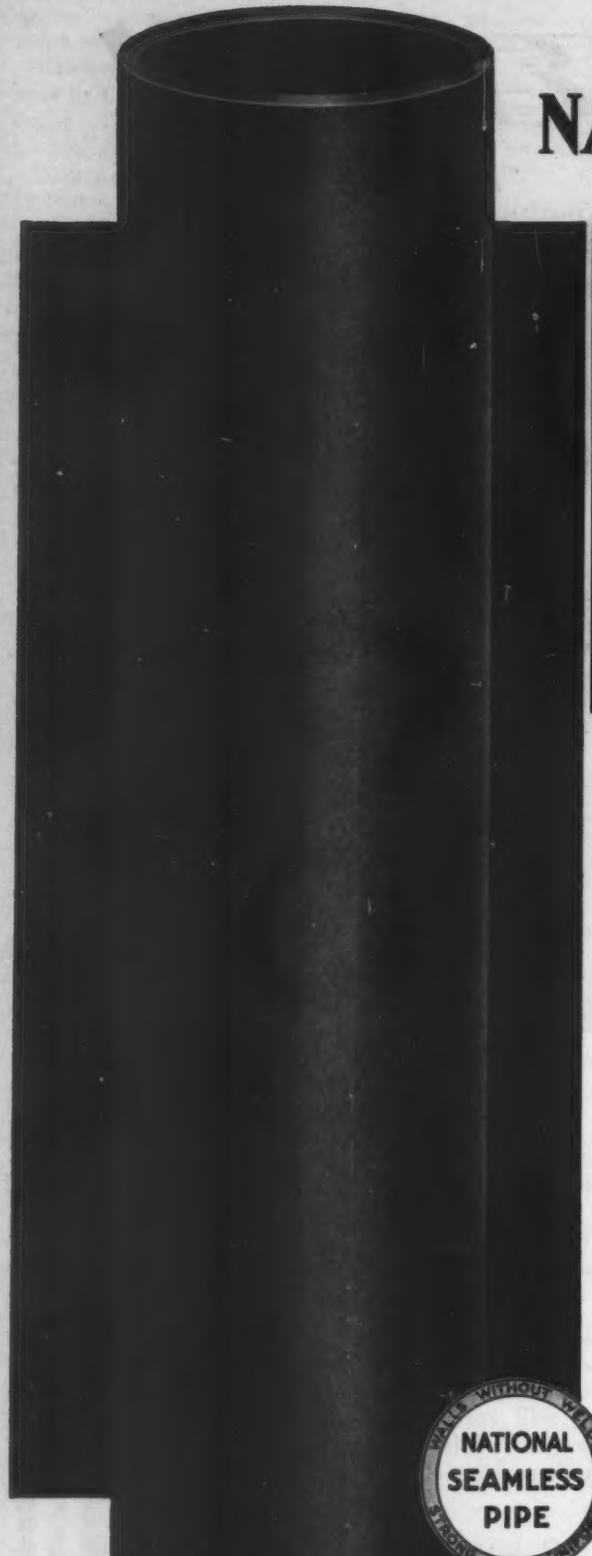

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THE MONTH IN REVIEW

Four Vice Presidents Elected by Institute

DIRECTORS of the American Petroleum Institute at the fourteenth annual meeting last month in Chicago had only the vice presidents and other officials to elect because President Axtell J. Byles was elected for 1934 when he accepted the place at the mid-year meeting in Tulsa last June.

The vice presidents chosen are: K. R. Kingsbury, vice president at large (re-elected); Charles F. Roeser, vice president for production (re-elected); Howard Bennette, vice president for refining, and C. E. Arnott, vice president for marketing.

W. R. Boyd, Jr., executive vice president, was re-elected, as were H. L. Pratt, treasurer, and Lacy Walker, secretary and assistant treasurer.

The board adopted a resolution to the effect that members of the executive committee may be represented by proxy. The executive committee consists of Axtel J. Byles, K. R. Kingsbury, Charles F. Roeser, Howard Bennette, C. E. Arnott, W. R. Boyd, Jr., H. L. Pratt, C. B. Ames, W. C. Teagle, H. F. Sinclair, F. A. Leovy, E. G. Seubert, J. Howard Pew and J. C. van Eck.

The membership committee selected for the next year consists of O. D. Donnell, R. A. Broomfield, Wirt Franklin, E. B. Reeser, and D. E. Buchanan. One of the first acts of the committee was to fix the dates of the next annual meeting, which will be held in Dallas, November 13, 14 and 15, 1934.

Membership of the board of directors was increased from 77 to 83 by election of E. L. Smith, Paul G. Blazer, Ralph T. Zook, W. S. S. Rogers, A. Jacobson and B. L. Majewski. The six directors-at-large elected annually by the board are J. Frank Drake, C. E. Arnott, W. S. Fitzpatrick, W. M. Irish and T. A. O'Donnell.

The following were elected to the general committee, division of refining, to serve during the coming year:

F. W. Abrams, Standard Oil Company of New Jersey, New York; L. S. Bale, Standard Oil Company (Ohio), Cleveland; C. B. Buerger, Gulf Refining Company, Pittsburgh; W. F. Burt, Socony-Vacuum Corporation, New York; L. R. Crawford, Producers & Refiners Corporation, Independence, Kansas; F. B. Dow, National Petroleum Association, Washington, D. C.; C. L. Henderson, Vickers Petroleum Company, Wichita, Kansas; F. E. Holsten, Barnsdall Refineries, Tulsa; E. W. Isom, Sinclair Refining Company, New York; Emby Kaye, Skelly Oil Company, Tulsa; E. R. Lederer, Texas Pacific Coal & Oil Company, Fort Worth, Texas; Harry Logan, United Refining Company, Warren, Pa.; K. G. Mackenzie, The Texas Company, New York; F. E. Rice, Phillips Petroleum Company, Bartlesville, Okla.; J. Noel Robinson, Tide Water Oil Company, New York; A. S. Russell, Standard Oil Company of California, San Francisco; W. L. Stewart, Jr., Union Oil Company of California, Los Angeles; F. W. L. Tydeman, Shell Development Company, San Francisco; C. B. Watson, Pure Oil Company, Chicago, and R. E. Wilson, Standard Oil Company (Indiana), Chicago.

The following five members were appointed by President Byles: J. Howard Pew, Sun Oil Company, Philadelphia; W. M. Irish, The Atlantic Refining Company, Philadelphia; P. M. Miskell, Empire Oil & Refining Company, Tulsa; Paul G. Blazer, Ashland Refining Company, Ashland, Kentucky, and D. J. Moran, Continental Oil Company, New York.

HOWARD BENNETTE

G. H. COXON, a director of Steaua Romana, Bucharest, and manager of the manufacturing division of Anglo-Persian Oil Company, Limited, was visiting this country during October to investigate processes and plant systems which may be of interest to his companies in their operations.

Chemical Engineers Meet After Exposition

TO make it possible for its members conveniently to attend the Fourteenth Exposition of Chemical Industries, which is to be held at Grand Central Palace in New York, December 4 to 9, 1933, the American Institute of Chemical Engineers has changed the dates of its national meeting to the following week. The meeting of the Institute will now be held December 12, 13 and 14, instead of December 6, 7, and 8. The place of the meeting is unchanged, namely Roanoke, Virginia.

In announcing this revision of dates of the Roanoke meeting, Dr. John V. N. Dorr, president of the American Institute of Chemical Engineers, stated that it was the opinion of the officers and council of the institute that the decision to change the meeting to the following week was in the best interests of all concerned. Not only was it a measure of cooperation with the Fourteenth Exposition of Chemical Industries, but a matter of natural convenience to the members of the institute.

Fourteenth Exposition Of Chemical Industries

THE Fourteenth Exposition of Chemical Industries, Grand Central Palace, New York, December 4 to 9, 1933, is broadly classified to include: raw materials, finished products, machinery and equipment and educational exhibits. There will be sections devoted to materials handling equipment and to instruments of precision. Plastics will be the subject of a special group and some of the phenomenal new applications of these materials will be displayed for the first time.

The previous Chemical Exposition had as exhibitors, 358 of the nation's leading companies and organizations. The audience of 100,000 arrived from 789 cities and towns in 45 of the United States and from 100 cities and towns in 38 foreign countries. The fourteenth exposition bids fair to surpass all records in the successful series of these expositions which began in 1915. The large trade expositions in this country have been the market places not only for products and machines but also of ideas. They have provided the time and the place to stimulate invention, to improve the methods by which things are produced. There is a power of industrial acceleration which is generated when the leaders in a field as broad as the chemical industry bring together their best work and indulge in the

profitable practice of comparison and competition. This year at the Chemical Industries Exposition the alert open-mindedness of the audience, and the progressive spirit of the exhibitors will constitute a double force, not only in the chemical industry and its 40 sub-industries, but in the industrial life of the country as a whole.

Clay Treating Licenses Broadened

TO handle the expanded field of licensing brought about by its enlarged scope of licensing rights for three clay treating processes, already in extensive commercial use, Gray Processes Corporation has appointed Alco Products Company, and M. W. Kellogg Company as its exclusive sales agents.

In July of this year Gray Processes Corporation entered into licensing agreements with The Texas Company, Standard Oil Company (Indiana), Pure Oil Company, and Standard Oil Development Company. By these agreements Gray obtained the right to grant licenses under the clay treating processes of the four companies. This involved an extended scope beyond that of the company's original activities with respect to the Gray process alone.

Gray's licensing rights now cover the three processes known in the industry as Gray, Osterstrom and Stratford, and include the clay treating developments of the four companies participating in the July agreements plus those of other licensees, among whom are The Atlantic Refining Company, Continental Oil Company and Sinclair Refining Company. Each of the three processes is already in extensive commercial use for refining cracked gasoline. The unified licensing structure makes possible the selection and use by a refiner of whichever of these clay treating methods or combination of them is most suitable to his requirements, under a single license agreement and at a single royalty.

Committee D-2 On Petroleum Products and Lubricants

A RECENT publication brings together in convenient form the many A.S.T.M. standard and tentative methods of test pertaining to petroleum and the 1933 report of Committee D-2 on Petroleum Products and Lubricants. In all there are 51 test methods given, many of which have been approved by the American Standards Association and the American Petroleum Institute.

Revisions in several of the standard methods were adopted in 1933 by the society. These are given in their latest form and cover the following tests: cloud and pour points; viscosity, flash and fire points with open cup, and sulfur in petroleum oils heavier than illuminating oils. Tentative standards included, which were revised this year, cover the tests for penetration of greases and petrolatum and tests for bituminous emulsions.

Several of the methods were adopted

Winners of Trophy Awards in 1933 Petroleum Section Contest.
Awarded at 22nd Annual Safety Congress, Chicago, October 5, 1933.

Front row, left to right: A. J. Martinson, Union Oil Co., of California, Los Angeles, first award, Pipeline Division, Group A. J. H. Brown, Tidewater Oil Corp., Bayonne, N. J., first award, manufacturing division, Group A. O. D. Donnell, Ohio Oil Co., Findlay, Ohio, first award, producing division, Group A. R. A. Hartzell, Standard Oil Co., of Indiana, Chicago, first award, marketing division Group A. A. W. Breeland, Lone Star Gas Co., Dallas, Texas, first award, natural gasoline division, Group A.

Back row, left to right: R. A. Gawthrop, Hope Construction & Refining Co., Pittsburgh, first award, production division, Group B. C. L. Hightower, Texas & Pacific Coal & Oil Co., Thurber, Texas, first award, marketing division, Group B. C. L. Bushnell, Ajax Pipe Line Co., Muskogee, Okla., first award, pipeline division, Group B. E. J. Senne, Socony Vacuum Oil Corp., for General Petroleum Co., of California, Los Angeles, first award, natural gasoline division, Group B. V. R. Currie, The Texas Co., for Indian Refining Co., Lawrenceville, Ind., first award, manufacturing division, Group B.

Designed for Processing Pennsylvania Crude to Produce:

1 Gasoline

2 Naphtha

3 Kerosene

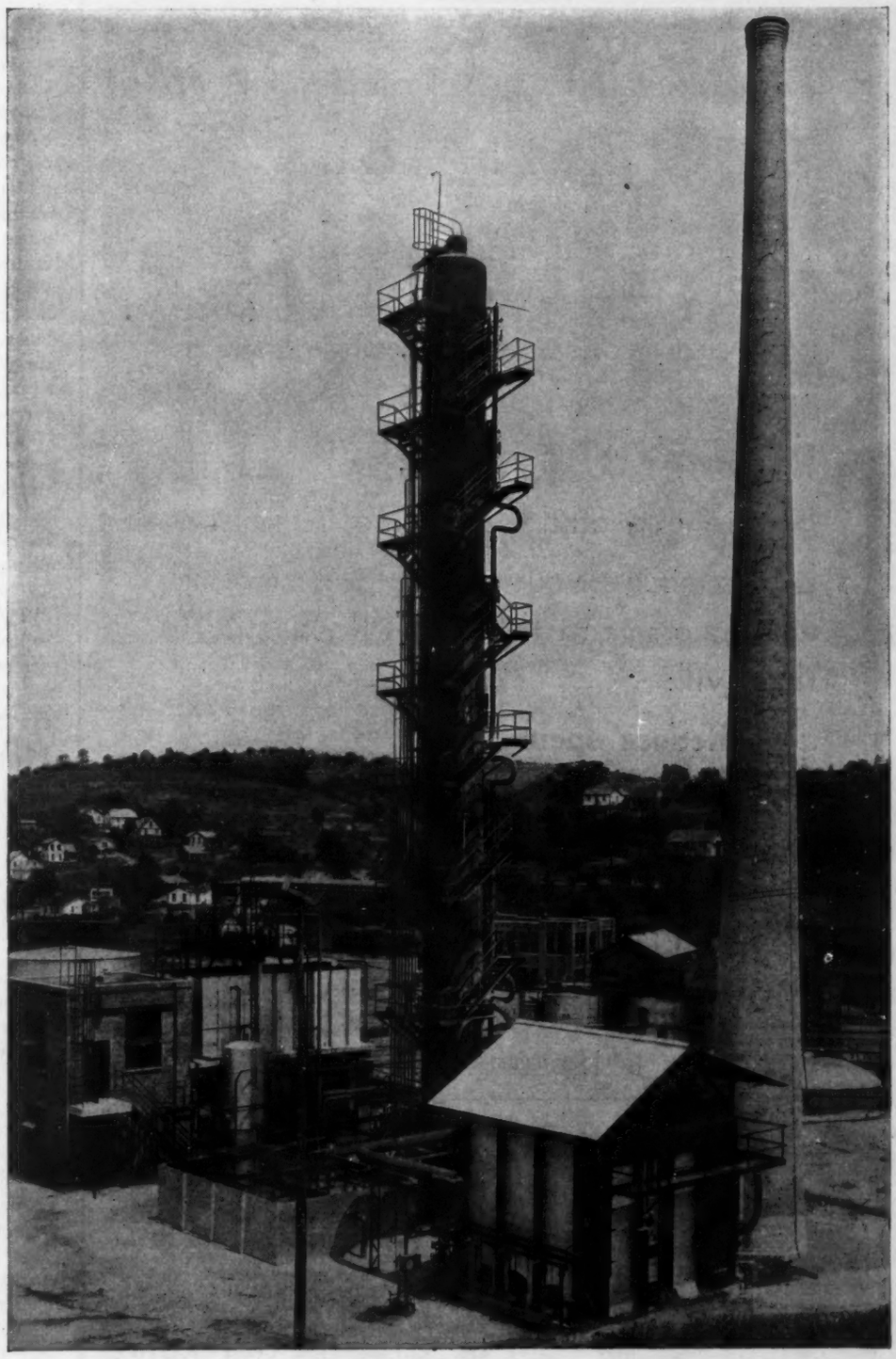
4 Furnace Oil

5 Gas Oil

6 Pressable Wax Distillate

7 Slop Wax

8 Steam Refined Bottoms



IN addition, this McKee Distillation Unit has been operated successfully and efficiently for re-running *Long Residum Solution* to *Flash and Fire Specification Long Residum* and also for re-running *Long Residum Solution* for Neutrals and *Specification Bright Stock.*

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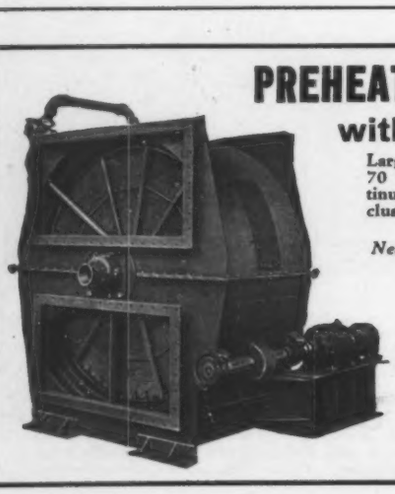

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as standard this year, having previously been approved as tentative. These new standards involve the following tests: dilution of crankcase oils, distillation of crude petroleum, gravity, precipitation number of lubricating oils, methods of sampling petroleum and its products, loss on heating of oil and asphaltic compounds, and tests for electrical insulating oils.

Other widely used standard tests which are given in the book cover determination of bitumen; burning qualities of kerosene, mineral seal and long-time burning (for railway use) oils; carbon residue; color of lubricating oils, petrolatum and refined oil; float test for bituminous materials; and specific gravity of asphalts, road oils and tars, asphalt cements, etc.

Other tests relate to analysis of grease; melting point of paraffin wax and petrolatum; detection in gasoline of sulfur and its corrosive compounds; vapor pressure of natural gasoline; water in petroleum products, etc.

The 1933 report of Committee D-2 summarizes the changes recommended in the various test methods developed by the committee and includes an appended technical paper "A Procedure for Measuring Bleeding of Cup Greases."

The complete publication aggregates 292 pages. Copies are available in heavy paper cover from A.S.T.M., 1315 Spruce Street, Philadelphia, at $1.25 per copy.

A. S. T. M. Symposium on Motor Lubricants

THE seven technical papers and extensive discussion which make up the 121-page Symposium on Motor Lubricants, being distributed now in book form by the American Society for Testing Materials, were prepared and presented at a joint meeting of the A.S.T.M. and the Metropolitan Section of the Society of Automotive Engineers, held in New York last spring. The symposium was arranged by A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. The subjects covered include service requirements, viscosity, carbon deposits, oil consumption, service changes, engine design as affecting oil performance, etc.

Many of the problems discussed in the symposium are controversial. Participation in the discussion by outstanding technologists in the petroleum and automotive industries resulted in a general advancement toward agreement on some of these factors. The authors used diagrams and tables extensively to give a quick and clear presentation of information.

Copies of the symposium are available in heavy paper binding at $1.25, or in cloth binding at $1.50, from A.S.T.M. 1315 Spruce St., Philadelphia, Pa.

Chlorex Solvent Process Licensed

THE Chlorex solvent refining process is to be licensed under patents and patent applications owned by Standard Oil Company (Indiana) and Mid-Continent Petroleum Corporation, and no royalty or license fee will be charged for the use of the process. Carbide and Carbon Chemicals Corporation, New York, it is understood, will license the process, and will furnish the Chlorex, which is the registered trade name for dichlorethyl ether. Licensees will sign contract for their supply of Chlorex to operate the process with Carbide and Carbon Chemicals Corporation, which concern is the sole manufacturer of the solvent and controls the patents under which it is manufactured. The contract assures the manufacturer that the solvent will not be purchased by the process owner in any case except from Carbide and Carbon Chemicals Corporation. The company states that considerable further reduction in operating costs has been made.

Shell Elects Taylor and Watson Vice Presidents

J. F. M. TAYLOR and J. W. Watson, both of St. Louis, were recently elected vice presidents of Shell Petroleum Corporation. Mr. Taylor, who has been general manager of manufacturing for the corporation since last December, will continue in charge of all manufacturing activities as well as of the related technical research and development work of the company. He has been with the Shell organization since 1921 and was superintendent of its Wilmington and Dominguez, California, refineries for several years.

Mr. Watson will act in a general capacity and as treasurer and comptroller in charge of all financial accounting, general personnel and stores matters of the corporation and its affiliated companies. He came to the corporation from California three years ago, was appointed comptroller January 1, 1932, and treasurer upon the death of T. F. Lydon last June.

Wall Washing in Cracking Operation

REFINERS operating cracking units in the Eastern and Appalachian districts cracking such distillates as gas oil, kerosene, and mixtures of the two from such crudes as the Pennsylvania grade, have found it expedient to wash the walls of the coke drums, soaking drums or reaction chambers. Heated oil and vapors leaving the furnace at the operating temperatures and entering the drums has a decided tendency to deposit coke as it

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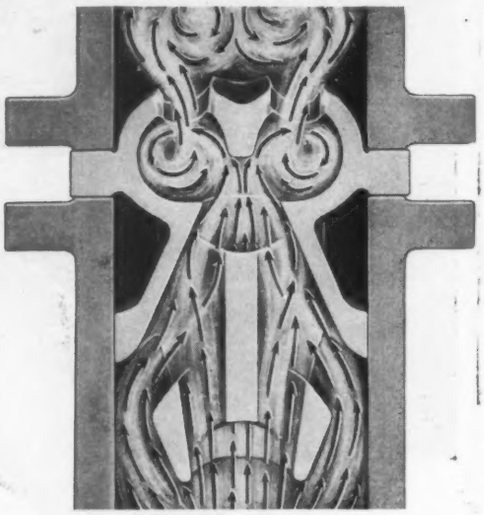
strikes the sides of the chamber. This continued cracking has formerly been retarded by quenching with cooler oil at the point of exit from the still and in the transfer line. Pennsylvania operation in several units is different in that quenching is done by pumping the cooler oil through a circular perforated coil placed near the top and around the inner circumference of the drum. An additional "wall washing" coil is placed at a point nearer the bottom of the drum so that the sides may be washed at both points if necessary. This practice retards the formation of coke from the very mobile liquids entering the drum, and makes possible longer runs and less shut down time. Through keeping a flow of oil down the sides of the drum the heated oils and vapors coming from the furnace are sufficiently cooled to largely reduce the coke formation, and such coke as may be produced is washed to the bottom of the chamber. It is said that the introduction of "wall washing" results in somewhat lowered rate of throughput, but with increased operating time on stream the practice is advantageous.



Cross-Section, Duriron Mixing Nozzle Installed in Pipe

HE'S A GOOD MIXER

Yes he is, this Duriron Nozzle. No baffle system can equal him. And he gives you mighty little back pressure and small friction loss. Easy to install; nothing to wear out. Just slipped in between the pipe flanges like a gasket.

Inlet and outlet views of Duriron Mixing Nozzles. Installation is most simple . . . no moving parts to get out of order. Let our representative give you complete information about the pumps, valves, heating and agitating jets, pipe and fittings, as well as special apparatus we make for Refineries.

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DURIRON

A. S. T. M. Has New Issue of Book of Standards

AMERICAN Society for Testing Materials has issued its book of A.S.T.M. Standards, a triennial publication containing all of the standard specifications, methods of test, recommended practices and definitions formally accepted by the society. The 1933 issue is composed of two parts: Part I containing all A.S.T.M. standards covering metallic materials and Part II, all standards pertaining to non-metallic materials, the two parts aggregating 2300 pages. Copies of the books can be secured from society headquarters at 1315 Spruce St., Philadelphia, at the following prices: Either part, $7.50; both parts, $14.00. Petroleum and petroleum products are included in the 285 standards contained in Part II—Non-Metallic Materials.

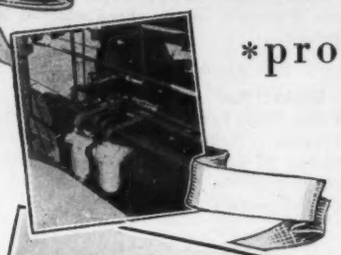
Pennsylvania College Petroleum Course

THE second registration for the fall and winter term of employee instruction in petroleum and natural gas, conducted by Pennsylvania State College mineral industries extension division, in cooperation with local school boards, the oil and gas industry and the American Petroleum Institute, was held early in October. At this time there were 22 registrations for the first year work, and 44 registrations for the second year work. Last year, at the close of the term 197 employees of oil and gas companies in Oil City, Franklin and Titusville received certificates after successfully completing the first year work.

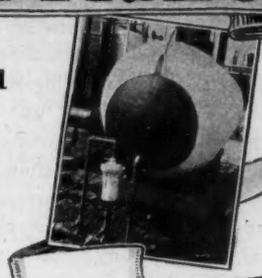
Among the group of instructors selected for the second year work is Dr. G. O. Ebray of Pennzoil Company and L. F. Walz, who were also instructors during last year's term. The course covers a period of three years work, comprising for the first year mostly fundamentals of mathematics, general chemistry, physics, electricity and shop instruction. The remaining courses available include unit processes, fluid flow, heat transfer, distillation, absorption, elementary organic chemistry, general geology, and natural gas and petroleum geology. There follows, natural gas and petroleum development, churn drilling, rotary drilling, shooting, cementing, development and safety work, also, petroleum production engineering, flowing, pumping, surface equipment, water flooding, repressuring, leasing, appraisals, transportation, storage, natural gas engineering, development and operation, gas measurement and pressure regulation, compression and transmission.

In addition to the regular instructors, the classes will hear lectures by engineers and technologists from the companies in

ARMSTRONG STEAM TRAPS



*process the steam that you use in processing



*Processing steam by an Armstrong steam trap means simply the automatic removal of the air and condensate from the steam, thereby permitting it to perform its maximum work of transferring heat.

Armstrong Machine Works
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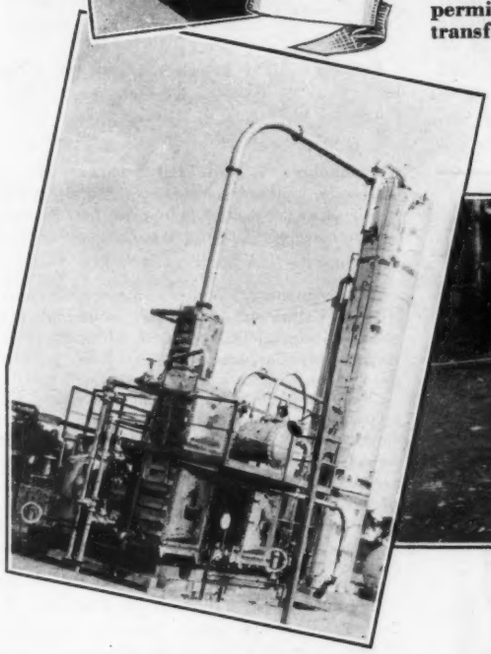
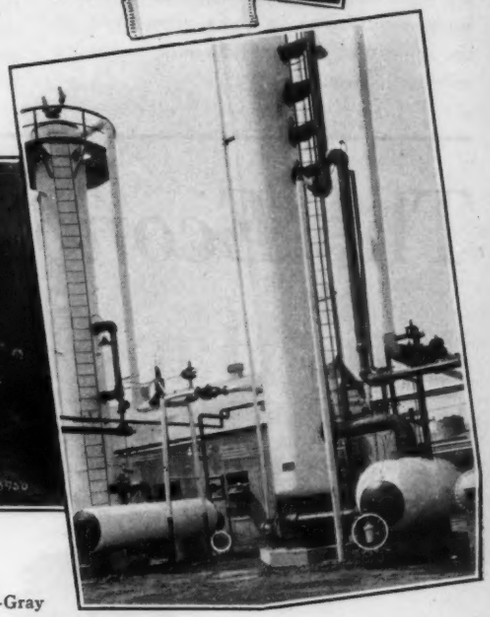
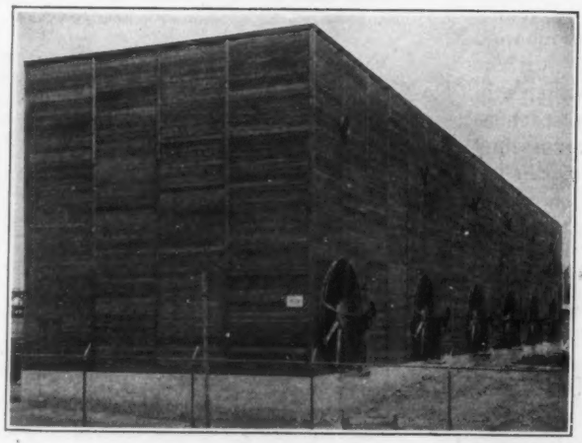
Three Rivers, Michigan





R11-Gray

IMPROVEMENTS



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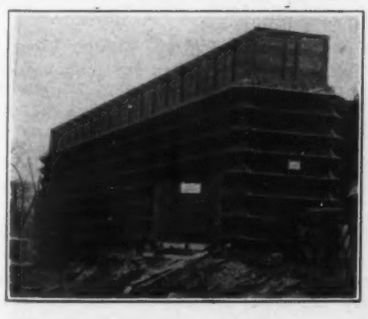


Above is a 7000 G.P.M. Marley forced draft refinery installation. Below is a 5000 G.P.M. Marley atmospheric deck type refinery installation.

MARLEY

COOLING SYSTEMS

the oil and gas industries and the petroleum industry equipment organizations. Early registrations give promise of a second term equally as successful as the term conducted last year. This was the first time the extension work was available and the second year's efforts are receiving the same full cooperation from oil, gas and equipment companies. The courses are not compulsory, the charges are nominal, and company employes have been enthusiastic in their acceptance of the opportunity offered by this university extension work.

▼ PLANT ACTIVITIES ▼

New Company: Pegasus Petroleum Products Company has been organized at Cleveland, Ohio, to establish and operate a refinery in the Cleveland territory. The organizers are George Ehrke, Bert D. Bradley and Faye Jenkins.

Operating: Omar Refining Company, formerly sales representatives for Garber Refining Company, Garber, Oklahoma, has purchased assets of that company at bankruptcy sale, and after rehabilitating plan resumption of operations early in November. Capacity 4500 barrels. Principle owners, Fred and Joe Whitaker, Tulsa.

Docks: Pan American Refining Corporation, Texas City, has let contract to Austin Bridge Company, Dallas, for construction of loading docks, to be built of creosoted timber, reinforced concrete docking, with berthing facilities for two tankers and one barge.

Refinery: Alkot Petroleum Company, Chase, Rice County, Kansas, building a 1000-barrel skimming plant.

Rebuilding: Triangle Oil Company, Venice, California, refinery, completely rebuilding 3000-barrel plant after destruction by fire. Contract to Southwestern Engineering Corporation.

Improvements: Walter Steiner Refinery, Hynes, California, installing additional fractionating equipment, contract to Southwestern Engineering Corporation..

Gasoline Plant: Smith Brothers Refinery Company will erect gasoline plant at McLean, Texas, to process gas from its own acreage around McLean (East Texas field). To consist of three absorption units and one distillation unit, capacity about 30,000 gallons. General contract to Tulsa Boiler & Machinery Company.

Improvements: Valvoline Oil Company, Butler, Pennsylvania, plant, has let contract to Foster Wheeler Corporation for one 2500-barrel crude oil distillation unit, one 2000-barrel solution redistillation unit, and one 700-barrel waste heat type special naphtha distillation unit. Additional improvements include new fullers earth burners, four automatic canning machines, additional tankage and new "Q" compound and Ethyl blending plants. Work to be completed in January. C. W. Luton, general manager.

Cracking Unit: Pennsylvania Oil Products Refining Company, Eldred, Pennsylvania, is installing a 500-barrel Vapor Phase Inc. type vapor phase cracking unit, to be in operation in November.

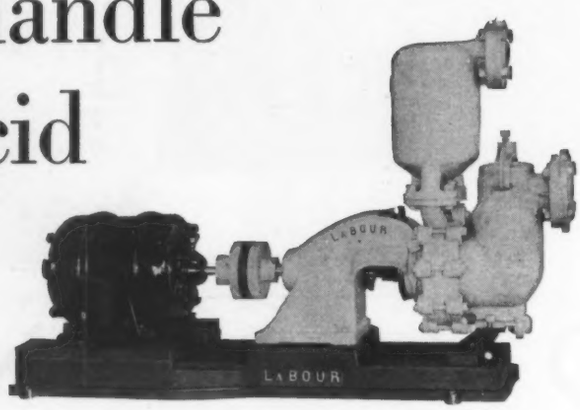
Operating: Olsen Refining Company, South Edmonton, Alberta, Canada, headed by Lars R. Olsen, started operating new 1000-barrel skimming plant early this month. Plant located on site of the old Alberta Refining Company.

Operating: Benco Refining Company, Three Rivers, Texas, recently organized by Henderson Coquat, completed and started operating 200-barrel refinery, producing lubricants, processing crude from Callihan field.

Canning Plant: Sinclair Refining Company, Houston plant, installing new canning plant annex, first unit a sheet iron building 40 feet by 100 feet, with 250-foot conveyor system from plant to shipside.

Centrifuge Plant: The Ultramar Petroleum Company, jointly owned by The Texas Company and Vacuum Oil Company, purchased a unit of several Sharples dewaxing centrifuges for installation at the company's Argentine, South America, refinery now nearing completion.

The *Economical* Way to Handle Acid



LaBour

Economy of time, of labor, and of maintenance costs follows the installation of LaBour pumps for handling corrosive liquids in refineries. Available in self-priming or gravity feed types, they start to move liquid promptly and certainly whenever they are called upon. The self-priming type provides extraordinary safety due to the fact that much of the line can be at less than atmospheric pressure. The hazards of bottom outlets and gravity feed are completely excluded.

The simple, rugged design of LaBour pumps makes them reliable. There is but one moving part —the impeller—and there are no valves, floats or other troublesome accessories. Time-out for repairs or replacement is down to the minimum when you use LaBour pumps.

Let the experience of the LaBour organization help you. This organization has long specialized in the problems involved in handling and conveying liquids in process plants. You will be under no obligation or cost in asking for full information.

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ORIGINATORS OF THE SELF-PRIMING CENTRIFUGAL PUMP

Enlargement: The Pennzoil Company, Oil City, Pennsylvania, has increased capacity of its dewaxing plant near Rouseville, through installation of a new battery of Sharples dewaxing centrifuges.

Synthetic Process: Anglo-Persian Oil Company is furthering its work in synthesis of higher molecular weight hydrocarbons from gases through installation of a semi-commercial plant, according to Dr. A. E. Dunstan, chief chemist, recently in America attending the meeting of the American Chemical Society, petroleum division, at Chicago.

Purchase: British-American Oil Company, Toronto, Ontario, Canada, has purchased the refinery and other physical assets of Sterling Oil Refineries, Moosejaw, Saskatchewan.

Solvent Refining: Gulf Refining Company, Philadelphia plant, has taken license under Duo-Sol patents and have commissioned Max B. Miller & Company to design a commercial plant for early installation.

Improvement: Roosevelt Oil Company, Mt. Pleasant, Mich., installing new still and fractionating column to replace equipment erected three years ago, and to improve efficiency, not increasing capacity above present 3000-barrel rating.

Refinery: Midwest Refining Company, Alma, Michigan (near Mount Pleasant), headed by B. J. Skinner, Grahd Rapids, is building a 1000-barrel skimming plant to process crude from the Porter Township pool.

Refinery: E. G. Guy proposes construction of a 1000-barrel skimming plant at St. Louis, near Mount Pleasant, Michigan, to run crude from the Porter Township pool. Guy was formerly associated with Old Dutch Refining Company and Roosevelt Refining Company in this area.

S-N Tri Process: Societe Franco Americaine de Raffinage, European subsidiary of Standard Oil Company of New Jersey, is erecting a Separator-Nobel centrifugal dewaxing plant, 400 barrels lubricating distillate per day. The plant employed five S-N separators, and used trichloroethylene as a solvent.

Rebuilding: Taxman Refining Company, Wichita Falls, Texas, is reported planning reconstruction of facilities recently destroyed by fire.

Refinery: Consolidated Petroleum Distributors Company, San Francisco, reported planning erection of modern refinery on Rough and Ready Island, Stockton, California.

Recovery Plant: Standard Oil Company of Louisiana at Baton Rouge, erecting new gasoline absorption plant, work being done by day labor.

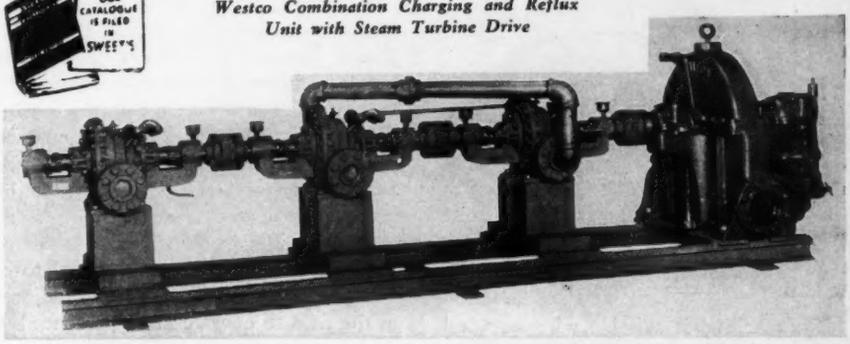
Gasoline Plants: Tall Pines Natural Gasoline Corporation to erect natural gasoline plants near London, south part of East Texas field, and another to be located in the northern section of the field. Company organized by Sidney Fitzner, Robert Kemp, and associates, Tyler, Texas.

Gasoline Plant: Cargray Corporation, Pampa, Texas, has started operating its 20,000-gallon natural gasoline plant, and is delivering production by pipe line to Phillips Petroleum Company.

Podbielniak Laboratories Moved to Chicago

"WHEN in Chicago come and see us at '222'" was the greeting on the clever card mailed to those in attendance at the recent conventions of the American Chemical Society and the American Petroleum Institute by Podbielniak Industrial Research and Analytical Laboratories, which recently established headquarters at 222 East Superior St., Chicago (across the street, by the way, from the rather famous "225 Club"). In these new headquarters the Drs. Podbielniak have arranged a complete display of the low and high temperature precise fractionating apparatus and other products which is well worth inspecting, as is the well equipped laboratories on the same floor. The Chicago laboratories are giving analytical and consulting services, and are equipped for glass blowing work of all kinds and for the construction of special scientific apparatus. Dr. Podbielniak still maintains his offices at 112 N. Lansing St. in Tulsa.

Westco Combination Charging and Reflux Unit with Steam Turbine Drive



HIGH PUMP EFFICIENCY

- stop hoping for it - it's HERE

LARGE capacities. Small capacities. Westcos handle either one against high pressures — and do it with high efficiency. That's because this modern pump's application of centrifugal force *is different.* Another feature is Westco's ability to deliver multi-stage performance with only single stage construction. This naturally eliminates the necessity for costly multi-stage construction.

Westco handles light, volatile, gasoline, propane etc. of 100° to 130° A. P. I. against pressures up to 300 lbs. —and does it without vapor binding.

Wide Operating Range

Westco automatically adjusts itself to any fluctuations in the operating pressure. An even, non-pulsating flow is assured at all times. By increasing the efficiency of the towers, this characteristic makes Westco the ideal pump for feed and reflux duty on high pressure stabilizers.

Only one moving part—the Impeller, which operates *without metal-to-metal contact inside the pump chamber.* This extreme simplicity means trouble-free operation, minimized maintenance and compactness.

Send for Details!

Without obligation, we'll gladly send you complete details that tell how and where you can apply Westco's valuable advantages in your own Refinery or Natural Gasoline Plant. Just fill in the coupon below and *mail it today.* *WESTCO PUMP CORPORATION,* Davenport, Iowa. *Branches:* New York, Philadelphia, Chicago, Los Angeles and San Francisco. *Representatives in 50 Principal Cities.*

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Davenport, Iowa, Dept. N-11.

Gentlemen: Without obligation, please send Performance and Selection Tables for Westco Turbine Charging and Reflux Pumps.

Name .....

Address .....

City .....

State .....

WESTCO *TURBINE* PUMPS

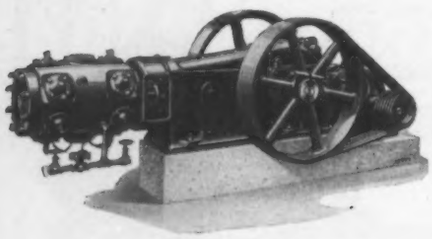
HIGH PRESSURE - - - SINGLE STAGE

New Equipment for the Modern Plant

Single Stage Compressor

INGERSOLL-RAND COMPANY

Ingersoll-Rand Company, 11 Broadway, New York City, is offering a new type, single-stage, belt-driven compressor which was designed for heavy-duty service. It is designated the Class ES, and has one horizontal, double-acting cylinder which operates at moderate speeds. It is available in sizes from 10 to 125 horsepower, and for discharge pressures from five to 150 pounds.



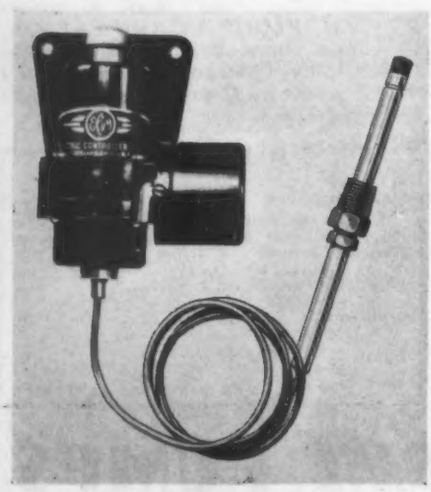
Ingersoll-Rand Compressor

The machine is suitable wherever full-load, continuous service is required, and wherever power cost is an important consideration. It was designed for use as a stand-by for large compressors where the maximum capacity is not always needed, for isolated plants, where oil in the discharge line is objectionable, where a future change in pressure conditions may call for a change in cylinder size, and for handling poisonous gases.

Bearing Thermostat

ELECTRIC CONTROLLER & MFG. CO.

Electric Controller & Manufacturing Company, 2700 East 79th Street, Cleveland, Ohio, announces the EC&M Bearing Thermostat. This thermostat was designed to protect motor-driven and other types of rotating machinery from breakdowns due to overheated bearings. It is well suited for use in connection with automatic pumping stations, motor-driven fans, automatic electric substations, conveyor systems, refrigerating machines, compressors, etc.



Bearing Thermostats

This thermostat is of simple construction and is compact. It consists essentially of a sealed metallic bulb, containing a volatile liquid which is connected directly or through flexible copper tubing to a one-piece seamless metal bellows. The electrical contacts are silver and are of double break, quick-make and quick-break construction. A rise in temperature of the bearing of a rotating machine causes the volatile liquid to expand and generate pressure in the bulb. The pressure in turn is transmitted to the bellows, causing the contacts to open and shut down the machine.

Bearing thermostats are shipped to operate at 100° C. They can be adjusted in the field to operate at 15 percent above or below this value. Proper operation is obtained with the bulb or relay installed in any suitable position—upright, horizontal or inverted.

Furnace Draft Regulator

CARRICK ENGINEERING COMPANY

Carrick Engineering Company, Michigan City, Indiana, announces Type HF-1 Furnace draft regulator for boilers up to 200 horsepower. It is used primarily to control the combustion chamber draft in the boiler plant, and while similar in design to the Carrick HF-2 regulator, the new design is intended for service where greater power is required. The principle

of the Carrick draft regulator is that of a beam scale. The draft is automatically "weighed," until the amount of draft for which it has been set is secured, thereafter this draft is automatically and accurately maintained in "balance" by the regulator. A bell, suspended from a beam, immersed in an oil seal, is the means used to measure the draft. The draft is applied to the under side of the bell, above the oil line. No diaphragm is used, thus eliminating maintenance at this point and assuring desired results.

Hydrometers

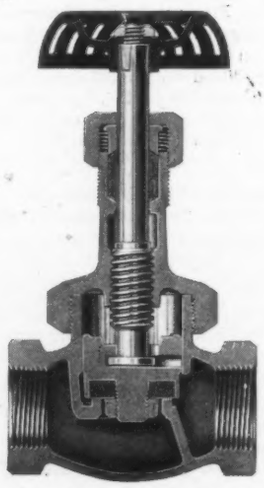
C. J. TAGLIABUE MFG. COMPANY

C. J. Tagliabue Manufacturing Company, Brooklyn, New York, announces the development of a new line of TAG certified hydrometers. The instruments are supplied in plain and combined forms and are said to be superior to the older models in accuracy, convenience and constructions. The metal thermometer scale, which is a new development, is much longer and the wide graduations allow temperature determinations to be made with much greater accuracy. All instruments are supplied with a signed accuracy certificate of guarantee. The design includes a stream-line tip, to permit of easy cleaning, solid metal ballast instead of mercury or lead shot, a metal scale, 25 percent longer than ordinary paper scales, a red stripe tube of large diameter, yellow scale with black figures to permit easy reading, a double gravity scale to permit reading from any position.

Disc Valve

THE LUNKENHEIMER COMPANY

The Lunkenheimer Company, Cincinnati, Ohio, has announced a non-metallic disc bronze valve suitable for 150 pounds steam pressure and 300 pounds gas-liquid pressure.



Lunkenheimer Valve

The valve has a deep non-heat malleable iron hand-wheel with an easy grip. The stuffing box is extra deep and holds a generous supply of packing. Bronze gland in all sizes has hexagon head to facilitatel removal. The stem is made of rolled bronze and is unusually heavy. Long threads are fully engaged with bonnet thread when valve is closed. Shoulder above the stem thread seats tightly against

bottom of stuffing box making the valve repackable under pressure.

The one piece bronze bonnet screws over the sturdy body neck and the flat face bonnet joint is easy to clean. The body is well proportioned with full flow areas. Exceptionally heavy body neck prevents distortion and makes a secure bonnet connection. Long pipe threads provide leak tight connections.

The disc holder is of the slip-on type—a turn or two of the handwheel before removing the bonnet prevents disc holder from dropping off. Extra long guides insure accurate seating. Lugs are provided for wrench hold when removing disc; the beveled extension protects spud threads. Non-metallic discs are produced to Lunkenheimer standards— for maximum life. Three basic types are offered —steam; hot water; cold water, gas and air.

Lunkenheimer Bronze "N-M-D" valves are regularly available in several designs —globe, angle, horizontal and angle check, spring check for compressor service and quick operating.

Taps and Dies

NATIONAL ACME COMPANY

National Acme Company is offering a complete line of circular chaser receding taps, ranging in size from four to 13⅜ inches, and a series of expanding dies,

Die Head

also employing circular chasers from one to 13⅜ inches. Larger sizes are available upon application.

Collapsible Tap

The fundamental idea back of the circular chaser, which resembles a circular forming tool, is to present a large amount of steel back of the cutting edge. This

PRECISION VALVE ACTION

. . . Because the Taylor Dubl-Response Unit is *mounted on the valve.*

NOT ONLY does the Taylor Dubl-Response System provide a sliding adjustment for sensitivity or throttling range (an exclusive feature).

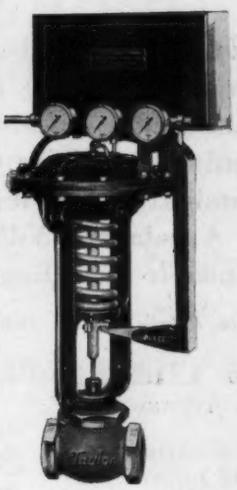
Not only does it offer an automatic "reset" for load changes.

But more important, it compels the valve to respond *precisely* to commands from the control instrument. It assures instant and automatic action.

There was a reason for mounting the Taylor Unit on the valve! It is the one way to get *precision valve action.*

That is why the Taylor Dubl-Response System will outperform all other regulators on difficult temperature, pressure or flow control installations.

Send for "Precision Control," a special bulletin that tells all about this recent development of Taylor engineers. A Taylor representative will be glad to discuss a specific application to meet your particular needs. Address Taylor Instrument Companies, Rochester, N. Y., or our branch office in Tulsa, Oklahoma.



Taylor Dubl-Response Control Unit in position on a Taylor Evenaction Diaphragm Valve.

SPECIAL TAYLOR NEWS FLASH

Beginning October 1st all Taylor Temperature and Pressure Recorders and Recording Regulators will be shipped in a new die-cast case originated by a noted American product designer. This modern case, with many distinct Taylor improvements in the mechanism of the instrument inside, represents another step in the effort to provide industry with the most up-to-date temperature and control equipment. More later.

Tycos Taylor

Indicating Recording • Controlling

TEMPERATURE *and* PRESSURE INSTRUMENTS

*The name Taylor now identifies our complete line of products, including Tycos instruments

HYDROGENATION

By CARLETON ELLIS

Starting with a clear account of the principles of hydrogenation catalysis, this book covers systematically the scientific work on the hydrogenation of individual organic compounds, and then presents exhaustively all the details of the industries themselves. The information in these pages has been compiled by the author over the many years of his career in this field. The researches, patents and inventions that have made him an outstanding authority on the subject of industrial hyrogenation tend to prove the value to you of this great book which has just been completed by the addition of new, hitherto unpublished information.

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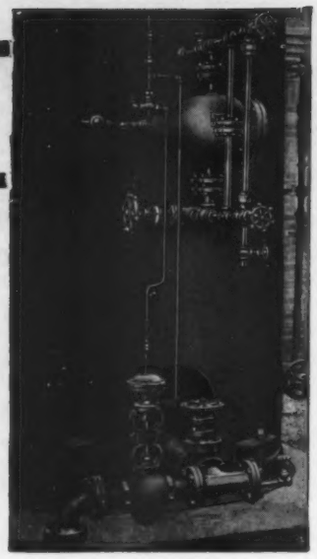
The Vigilant Liquid Level REGULATOR



For Towers, Tanks, Stills, etc., for temperatures up to 800 degrees F. — For continuous or intermittent, inlet or outlet flow: This C-F Regulator maintains correct level under any load. A balanced V-Port Valve gives dependable throttling action.

Complete details gladly furnished.

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ROTO TUBE CLEANERS

built for* Oil Still Tubes, 3″ I.D. *and up

Roto Special 6-way Drill Head and Universal Joint.

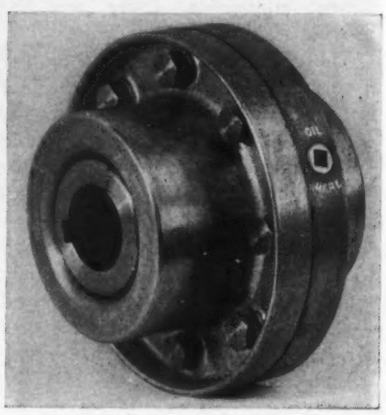
Roto Combination Head and Universal Joint.

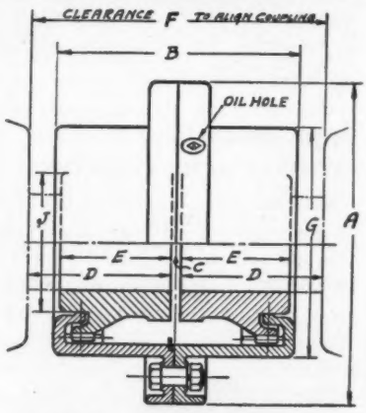
THE ROTO COMPANY

Sussex Avenue and Newark St., Newark, N. J.

dissipates cutting heat and affords support to the cutting edge.

The chasers are ground on the form and mounted rigidly on a massive steel block, being inclined on the surface of the block in such a way that the successive chasers describe a helix on the pipe.





All Dimensions are given in inches

SIZE NO.	MAX BORE	HP PER 100	MAX R.P.M.	A	B	C	D	E	F	G	J	SHIPPING WEIGHT IN LBS.
1	1	2	3600	4 1/16	3 1/8	1/2	1 15/16	1 3/16	4	2 3/4	1 1/2	5 1/4
1 1/4	1 1/4	3.5	3600	5 1/16	3 1/16	1/2	2 1/16	1 1/16	4 7/16	3 1/4	1 3/8	7 1/4
1 1/2	1 1/2	6	3600	5 7/8	4 1/2	1/2	2 7/16	1 5/8	5	3 1/16	2 1/8	10 3/4
1 3/4	1 3/4	10	3600	6 7/8	4 5/8	1/2	2 13/16	2 1/16	5 3/8	4 1/2	2 9/16	15
2	2 1/4	15	3600	6 3/4	5 3/8	1/2	3 1/8	2 7/16	6 3/8	4 11/16	2 3/8	17 3/4

Fasts Light Duty Coupling

Light Duty Coupling

BARTLETT HAYWARD COMPANY

Bartlett Hayward Company, Baltimore, Md., announces a new series of Fast's self-aligning couplings in sizes ranging from one-inch to 2⅛-inch for horsepowers from two to 25 per 100 r.p.m. and a minimum speed of 3600 revolutions. The new Fast's light duty coupling was developed to meet the need for an inexpensive trouble-proof coupling from drives from one to 25 horsepower and retains all of the features of the company's heavy duty forged steel coupling except that the casing is made of die cast metal. The performance of this coupling is guaranted for all horsepowers and speeds as given in the accompanying illustration. The light coupling

has been under test in various applications the past three years, giving satisfactory performance.

Mercury Switch

THE BROWN INSTRUMENT CO.

The Brown Instrument Company, Philadelphia, announces the use of mercury switches to eliminate open make-and-break contacts controlled by the pen arm as a feature of the new automatic control thermometer. In this controller, every six seconds a motor-driven control table determines the location of the pen on the chart in reference to the control setting, and tilts the mercury switch from one side to the other if the temperature has changed.

In this system the measuring mechanism is free to position itself, unhampered by the control mechanism. There is no excess weight on the pen, no distortion of the pen arm, no restriction of the pen movement. The making of control contacts is not dependent upon friction because the switch is positioned with a positive action by the electric motor. Contacts are unaffected by vibration because the mercury switch is mechanically locked in place until a different position is required. Corrosion and dirty contacts are eliminated as the switch is sealed in glass. These mercury switches have capacities up to 15 amperers at 110 volts (10 amperes at 220 volts), thus eliminating external relays in most applications.

This control mechanism is adaptable to one, two, or three-contact control (or signalling) systems. In a two-record controller, each control mechanism can control a separate device, each may be used independently of the other, and each has its own control index.

This new Brown controller is also supplied as a time-temperature cycle controller. A transparent cam placed over the chart is used to shift the control index in relation to time. As the cam rotates with the chart, the control point is moved to new positions, depending upon the shape of the cam. A different cycle of control operations can be obtained by simply cutting a new cam.

All models of this controller are furnished in circular 10-inch or 14-inch dustproof cases, and offer all the outstanding features of the new Brown Recording Thermometer and Pressure Gauge, introduced two months ago. For example, the measuring mechanisms are rugged and powerful; electric chart drive is standard at no extra cost; cases are back or bottom connected and interchangeable for flush, front-of-board or wall mounting.

Contact Gauges

THE HAYS CORPORATION

The Hays Corporation, Michigan City, Indiana, announces a complete line of compact electric contact gauges for draft, pressure and differential draft or pressures in ranges between one tenth-inch water and 100 inches water and employing the Hays dry type actuating unit. These gauges are offered for either high or low contact service, or both, equipped with closed mercury switches or open contacts, depending upon the service conditions. The small cast aluminum case can accommodate either one or two single or double contacting units, or a single or double contact unit and a second unit which indicates on a three-inch scale the pressure of draft being measured. For differential draft or pressure the case is made air-tight. Sensitive adjustments across the range selected are provided in a simply and accessible manner. These fully developed contact gauges are offered for all manner of applications where it is desired to sound an alarm or light a bullseye when certain pressures reach a predetermined high or low point, as in drying, ventilating, dust collecting and similar systems. Used in conjunction with intermediate devices offered on the market the gauges are employed to open and close dampers, start and stop fan or vibrator motors, and in similar applications within their pressure range.

TROUBLE STARTS IN UNFILTERED PIPE LINES

A Protectomotor Air Filter on the compressor makes it run 3 to 5 times longer before overhauling is needed. But it is not sufficient protection for tools at the other end of line.

There is always some oil, water, scale, rust and grit in compressed air lines, to play havoc with the tools and spoil costly work.

Therefore, every pipe line should have a separate Protectomotor Pipe Line Air Filter to remove all foreign matter from the compressed air. This filter insures delivery of dry air that is 99-9/10% clean, for paint spraying, cleaning operations, agitating liquids, chemical processes, and the like. It also increases the life of compressed air tools, reduces maintenance costs and increases their efficiency.

The Protectomotor is a dry filter and is easy to clean.

Write for catalog and particulars regarding our 30-day free trial offer.

Staynew Filter Corp.
21 Leighton Ave. Rochester, N. Y.

PROTECTOMOTOR
REG. U.S. PAT. OFF.
99 9/10 Per Cent EFFICIENT
AIR FILTERS

Now Is the Time

TO RE-COVER YOUR FILTER LEAVES WITH LONG LASTING

SMOOTH-TEX
METALLIC FILTER CLOTH

You can do the work, using our improved method and fittings or we do it for you.

Write to-day

MULTI-METAL WIRE CLOTH CO., INC.
795 EAST 139TH ST.
NEW YORK, N. Y.

"PROVE IT," said

the refinery superintendent to an Atlas Salesman. "How can you prove that the CAMPBELL Boiler Feed Water Regulator is the very regulator we need on our refinery boilers?"

"Very easy," replied the Atlas man. "Here is a list of big refiners who installed the CAMPBELL, tested it thoroughly, and then ordered *more* CAMPBELL'S. We are receiving repeat orders continually."

Would you too, Mr. Refiner, like to see that list? Mailed on request.

Cost of installation is remarkably low. No floats. No thermostats. No generators. No moving parts in the Regulating Element. Nothing to get out of order.

We make Regulating Valves for Every Service. See partial list in coupon below. Make it a habit to put all of your regulating problems up to Atlas.

Patented

ATLAS VALVE COMPANY
REGULATING VALVES FOR EVERY SERVICE
275 South St., Newark, N. J.

Clip this coupon to your letterhead, check the items in which you are interested, and we will gladly send prices and data. No obligation.

- ☐ The Campbell Boiler Feed Water Regulator
- ☐ Reducing Valves
- ☐ Damper Regulators
- ☐ Exhaust Control System
- ☐ Temperature Regulators
- ☐ Pump Governors
- ☐ Float Valves
- ☐ Swing Joint Fittings
- ☐ Bronze Unions
- ☐ Thermostats
- ☐ Balanced Valves
- ☐ Control Valves

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Webster Engineering Co.
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Worthington Machinery Corp.

Items About Manufacturers

T. Shriver & Company, Harrison, N. J., manufacturers of filter presses, announces appointment of The Merril Company, engineers, 343 Sansome Street, San Francisco, as sales representatives in the Rocky Mountain and Pacific Coast areas.

California Chemical Corporation, 220 Bush Street, San Francisco, announces appointment of Sidney P. Armsby, as assistant sales manager. For the past two years Mr. Armsby has been in private practice as technical consultant on industrial raw materials and processes in the Gulf Coast area, Texas, and prior to that was with Haden Lime Company, Houston.

Struthers-Wells-Titusville Corporation, Warren, Pa., has opened an office at 2325 Gulf Building, Houston, under the direction of O. E. Berg, district sales manager.

Sarco Co., Inc., New York City, has appointed the following two new representatives: H. C. Freeze, 811 East Armour Boulevard, Kansas City, Mo., and Colby Equipment Company, 136 N. Delaware Avenue, Indianapolis, Indiana.

The Duriron Company, Inc., Dayton, Ohio, announces the appointment of E. D. Brauns as manager of the company's Philadelphia office. He was for many years in the company's engineering department and general sales department in Dayton and for the past year has been in charge of the Boston office.

Blaw-Knox Company, Pittsburgh, announces appointment of John S. Conroe as a member of the process equipment department. He formerly was in the same line of work with Struthers-Wells-Titusville Corporation.

Steel and Tubes, Inc., Cleveland, Ohio, announces the following changes in personnel: A. V. Grove has been transferred from Cleveland to the Chicago office, in the sales department. R. E. Doyle is now sales correspondent in the Cleveland office, and J. F. Keeler is now sales engineer with headquarters in the Cleveland office.

Gray Processes Corporation announces appointment of Alco Products, Inc., and the M. W. Kellogg Company as its exclusive sales agents. The commercial processes affected are the Gray, Osterstrom, and Stratford, and include clay treating developments coming from seven large oil refining companies.

Northern Equipment Company, Erie, Pa., announces appointment of C. J. Gaskell Company, Memphis, Tenn., as representatives for Copes feed water regulators, differential valves, pump governors and allied equipment.

Podbielniak Industrial Research and Analytical Laboratories have established laboratories, offices and display rooms at 222 East Superior Street, Chicago. Complete exhibits of the Podbielniak low and high pressure precise fractionating apparatus are maintained for inspection.

Byron Jackson Company announces appointment of J. M. McGregor as sales engineer for Tulsa and vicinity. He formerly was president of Macadam Engineering Company, Tulsa.

Bastian-Blessing Company announces appointment of Charles W. Steele as sales manager of the "Rego" welding and cutting division of the company. M. H. Kauer has been made sales promotion and advertising manager.

Universal Oil Products Company has transferred Geo. W. Miller of the company's New York staff to Tulsa, where he is reported recovering from a severe illness of several months duration. Miller has seen many years service with Universal Oil Products Company in many parts of the world and his friends all wish him a speedy recovery.

WANTED—Engineer with knowledge orifice meters, flow controllers, etc., and experience their application in refineries; must be available for long distance traveling. Write, stating education, experience and salary desired. Box 1889, C/o Refiner, Houston, Texas.

STATEMENT OF THE OWNERSHIP, MANAGEMENT, CIRCULATION, ETC., REQUIRED BY THE ACT OF CONGRESS OF AUGUST 24, 1912.

Of The Refiner and Natural Gasoline Manufacturer, published monthly at Houston, Texas, for October 1, 1933.

State of Texas, County of Harris: ss.

Before me, a Notary Public in and for the State and county aforesaid, personally appeared A. L. Burns, who, having been duly sworn according to law, deposes and says that he is the Business Manager of the Refiner and Natural Gasoline Manufacturer and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management (and if a daily paper, the circulation), etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, embodied in section 411, Postal Laws and Regulations, printed on the reverse of this form, to wit:

1. That the names and addresses of the publisher, editor, managing editor, and business managers are:

Publisher: R. L. Dudley, 3238 Huntington Drive, Houston Texas.

Editorial Director: S. W. Robinson, 5002 Scotland, Houston, Texas.

Editor: Grady Triplett, 2202 Dunstan Road, Houston, Texas.

Managing Editor: J. Kent Ridley, 605 Sul Ross, Houston, Texas.

Business Manager: A. L. Burns, 4311 San Jacinto, Houston, Texas.

2. That the owner is: (If owned by a corporation, its name and address must be stated and also immediately thereunder the names and addresses of stockholders owning or holding one per cent or more of total amount of stock. If not owned by a corporation, the names and addresses of the individual owners must be given. If owned by a firm, company, or other unincorporated concern, its name and address, as well as those of each individual member, must be given.)

The Gulf Publishing Company, Houston, Texas; James Anderson, Houston, Texas; R. L. Blaffer, Houston, Texas; Mrs. Sarah Campbell Blaffer, Houston, Texas; A. L. Burns, Houston, Texas; J. F. Carter, Jr., Tulsa, Okla.; Dr. N. L. Dudley, Goose Creek, Texas; R. L. Dudley, Houston, Texas; W. S. Farish, Houston, Texas; Wm. V. Gross, Houston, Texas; Howard R. Hughes, Houston, Texas; Chas. H. Lane, Houston, Texas; Wallace E. Pratt, Houston, Texas; J. Kent Ridley, Houston, Texas; S. W. Robinson, Houston, Texas; Mrs. Maude G. Sterling, Houston, Texas; R. P. Swinsky, New York, N. Y.; O. R. Waller, Chicago, Ill.

3. That the known bondholders, mortgagees, and other security holders owning or holding 1 per cent or more of total amount of bonds, mortgages, or other securities are: (If there are none, so state.) None.

4. That the two paragraphs next above, giving the names of the owners, stockholders, and security holders, if any, contain not only the list of stockholders and security holders as they appear upon the books of the company but also, in cases where the stockholder or security holder appears upon the books of the company as trustee or in any other fiduciary relation, the name of the person or corporation for whom such trustee is acting, is given; also that the said two paragraphs contain statements embracing affiant's full knowledge and belief as to the circumstances and conditions under which stockholders and security holders who do not appear upon the books of the company as trustees, hold stock and securities in a capacity other than that of a bona fide owner; and this affiant has no reason to believe that any other person, association, or corporation has any interest direct or indirect in the said stock, bonds, or other securities than as so stated by him.

A. L. BURNS,
Business Manager.

Sworn to and subscribed before me this 29th day of September, 1933.

(Seal) J. KENT RIDLEY.

(My commission expires June 30, 1935.)